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ENVIRONMENTAL MONITORING NEAR INDUSTRIAL SITES

ACRYLAMIDE

$CH_2 = CHCONH_2$

MARCH 1978

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF TOXIC SUBSTANCES
WASHINGTON, D.C. 20460

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16. ABSTRACT

A sampling and analysis program was conducted to determine the levels of acrylamide in air, water, soil, and sediment around five industrial sites. The industrial sites included monomer producers, polymer producers, and polymer users.

Air was collected at the plant perimeters using Chromosorb 101 adsorption tubes. Soil samples were collected at the air sampling stations. Water and sediment samples were taken upstream and downstream and at the plant discharge.

No acrylamide was found in any of the air, soil, or sediment samples. Acrylamide was found in only one water sample. The level was 1,500 ppb. The identification of acrylamide was confirmed by GC/MS.

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ENVIRONMENTAL MONITORING NEAR INDUSTRIAL SITES ACRYLAMIDE $CH_2=CHCONH_2$

Contract No. 68-01-4115

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March 1978

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SECTION I

SUMMARY

The purpose of this program is to provide sampling and analysis capabilities to Environmental Protection Agency's (EPA) Office of Toxic Substances, so that the levels of suspected toxic substances in air, water, soil, and sediment at designated locations throughout the United States could be determined. The second task was the sampling and analysis for acrylamide (AA).

Methods for sampling and analyzing AA in air, water, soil, and sediments were developed or modified.

Air samples were collected on Chromosorb 101 and desorbed for analysis with methanol. Water samples and methanol extracts of soil and sediment samples were concentrated by evaporation prior to analysis. All extracts were analyzed by gas chromatography with nitrogen selective detection. The detection limits of environmental samples varied but were generally 0.2 $\mu g/m^3$ for air, 0.8 μg/liter for water, and 0.02 $\mu g/g$ for soil and sediment.

Five industrial sites were selected for sampling. Two plants were producers of AA, two plants were producers of AA and polyacrylamide and one plant was a polyacrylamide user. A sixth site, for which some results are reported, was sampled previously by another contractor.

A summary of the analytical results from the six industrial plants is given in Table 1. No AA was found in any air samples in either the vapor or particulate form. Acrylamide was detected in the aqueous discharge stream for one plant sampled by MRI. The level found was 1,500 ppb and the identification of AA was confirmed by GC/MS. No AA was detected in any of the soil or sediment samples.

Table 1. ANALYSIS DATA SUMMARY FOR TASK 2

Site	Process	Air ($\mu g/m^3$)[a]		Water ($\mu g/\ell$)[a]		Soil ($\mu g/g$)[a]		Sediment ($\mu g/g$)[a]	
		High	Low	High	Low	High	Low	High	Low
Hercules, Inc. Hopewell, Virginia	Producer of polyacrylamide	< 0.4-1.1		1,500	< 0.8	< 0.08		< 0.08	
Calgon Corporation Ellwood City, Pennsylvania	Producer of polyacrylamide	< 0.1		< 0.8		< 0.02		< 0.02	
Nalco Chemical Company Garyville, Louisianna	Producer of acrylamide and polyacrylamide	< 0.1-0.3		< 0.8		< 0.02		< 0.02	
American Cyanamid Fortier, Louisianna	Producer of acrylamide and polyacrylamide	< 0.1		< 0.8		< 0.02		< 0.02	
St. Regis Paper Company Sartell, Minnesota	User of polyacrylamide	-		< 0.8		-		-	
Dow Chemical[b] Midland, Michigan		< 1.0		25-125	< 25	< 0.02		-	

a/ When no acrylamide was detected, the detection limits have been given.
b/ Data from Reference 1.

SECTION II

INTRODUCTION

Acrylamide is the most important chemical in its group, being produced at an estimated rate of 63 million pounds per year as of 1976.[2] By 1978, all production should be based on catalytic hydration of acrylonitrile. The major producers of AA are American Cyanamid, Dow Chemical, and Nalco Chemical. The principal use of AA (~ 80%) is in the production of water soluble polymers, commonly called polyelectrolytes, flocculants, coagulants, thickening agents, retention aids, and drainage aids. Polymer production consumes nearly all AA production, much of it captively.

Acrylamide release to the environment could occur at monomer and polymer manufacturing sites, polymer application sites, and in transportation and handling. Air levels of AA would not be expected to be high due to its low vapor pressure of 0.007 mm Hg at 25°C. Aqueous discharge is a more likely route of release because of its extremely high water solubility (216 g/100 ml at 30°C). The only available monitoring data for the discharge of AA into the environment is from Croll's study in Great Britain during which levels as high as 42 µg/liter were observed.[3]

The environmental concern about AA results principally from its reported neurotoxicity. The acute LD_{50} in rodents[4] is 200 mg/kg; however, lower dosages have shown a cumulative toxicity.[5] The carcinogenicity of AA is unknown.

The remainder of this report describes the experimental methods employed, including the development of the sampling and analysis protocol, the selection of sampling sites, and a discussion of the monitoring results.

SECTION III

EXPERIMENTAL METHODS

SAMPLING PROTOCOL

Air Sampling

Air was sampled at four to five stations deployed upwind, downwind, and crosswind of the industrial sites. Duplicate samplers were placed at some of the stations. The sampling probes were set at 1.5 m above ground and air was sampled at 0.5 to 1 liters/min continuously for up to 24 hr.

The meteorological conditions that existed during each test were obtained from the nearest National Weather Station. Wind speed and direction, temperature, and precipitation were included in the data log.

The sampling train for the collection of AA from ambient air consisted of the following components assembled in the order given: (a) Millipore filter (AAWP03700), directed into the wind; (b) top Chromosorb 101 tube; (c) bottom Chromosorb 101 tube; and (d) 24-v DC vacuum pump.

The adsorbent tubes were constructed of glass and were 8 mm OD x 6 mm ID x 18 cm in length. Prior to use the internal surfaces were silanized. The tubes were packed with 0.7 g 60/80 mesh Chromosorb 101 that had been Soxhlet extracted with methanol overnight and then dried at 150°C for 4 hr under a stream of nitrogen. Minimum lengths of natural latex tubing were used to connect the sampling tubes to the pumps. No critical orifice was used as the pressure drop of the two tubes was sufficient to keep the flow rate under 1 liter/min. The actual flow in the field was monitored by calibrated rotameters every 3 to 4 hr. Power was supplied to the DC pump by two 24-v lantern batteries connected in parallel. The pump and batteries were encased in a cardboard-polystyrene shipping box which was then anchored at the sampling site. The reliability of this system has been demonstrated in previous OTS studies.[6,7] After sampling was completed, the tubes were capped with rubber slip-on septa, wrapped in aluminum foil and stored over dry ice for shipment to MRI. At MRI, the tubes were kept on dry ice until analyzed.

Water Sampling

Grab water samples at the outfalls were collected periodically during the air sampling period and composited in 1 gal. brown silanized bottles with Teflon®-lined caps. Upstream and downstream samples were collected concurrently. After collection the samples were placed on wet ice for shipment to MRI. At MRI, the samples were stored at 2 to 4°C.

Soil Sampling

Soil samples from the top 10 mm were collected at the air sampling stations and placed in 16-oz jars with aluminum foil-lined caps. The jars were then stored over dry ice for shipment to MRI. At MRI, the samples were kept on dry ice until analyzed.

Sediment Sampling

Sediment samples were collected by use of an Eckman dredge. The samples were placed in 16-oz jars with aluminum foil-lined caps. After collection the samples were placed and shipped on wet ice to MRI. At MRI, the samples were stored at 2 to 4°C.

ANALYSIS PROTOCOL

Air Samples

The Chromosorb 101 tubes were inverted and eluted with 10 ml of methanol. The methanol extract was then reduced in volume to approximately 0.5 ml by evaporation with a nitrogen stream. The Millipore particulate filters were extracted for 0.5 hr in an ultrasonic bath with 2.0 ml water.

The extracts were analyzed by GC with electrolytic conductivity (Hall) detection. The operating parameters were as follows:

Column: 1.8 m x 2 mm ID silanized glass
Packing: 80/100 mesh Chromosorb 101
Carrier: Helium at 30 ml/min
Reaction tube: Quartz with Ni catalyst, hydrogen at 35 ml/min
Electrolyte: 15% isopropanol/water, 0.5 ml/min
Injector temperature: 240°C
Column temperature: 220°C
Reaction furnace temperature: 820°C

A limited number of sample extracts were spiked with a known quantity of AA and reanalyzed. The results were used to establish limits of detection.

Water Samples

A 250-ml aliquot of the water sample was reduced in volume to about 10 ml by rotary evaporation. The sample was then further reduced in volume to 0.5 to 8 ml using a nitrogen stream. Any solid which formed during the evaporations was removed by centrifugation and extracted with methanol. The two fractions were analyzed separately by GC using the conditions outlined above. A limited number of samples were spiked with a known amount of AA and reanalyzed in order to establish the detection limits.

Soil Samples

Fifty-gram aliquots of the soil samples were extracted twice with 50 ml portions of methanol using ultrasonic agitation. The extracts were combined and evaporated to 5 ml. Any solid which precipitated was removed by centrifugation. Separate soil samples were dried at 110°C for 4 days in order to determine the moisture content. The sample extracts and spiked sample extracts were analyzed by GC using the conditions outlined above.

Sediment Samples

Sediment samples were first centrifuged to separate the water which was then removed. A 50-g sample was then prepared and analyzed in the same manner as the soil samples.

GC/MS Confirmation

Samples were confirmed by GC/MS using the same column and conditions described above. In order to eliminate a column bleed problem, the MS was operated in a high resolution selected ion monitoring mode. Although only one mass was monitored, the selectivity provided by high resolution MS virtually eliminated all compounds except a C_3H_5NO. The matching of retention times then confirms the presence of AA. The instrument operating conditions were as follows:

Instrument: Varian MAT 311A mass spectrometer with Varian 2700 gas chromatograph
Data system: Varian Spectrosystem 100 MS
Separator: Watson-Bieman
Mass monitored: 71.0371
Resolution: ~ 7,000
Ionization potential: 70 ev
Emission current: 1 mA
Multiplier gain: $4 \times 10^6 - 2 \times 10^7$

METHOD DEVELOPMENT FOR SAMPLING AND ANALYSIS

A protocol for sampling and analysis of AA was under development by Batelle Memorial Institute (BMI). The techniques which were proposed and used at one site are summarized in Table 2. Also included is the extent to which the method was validated. In discussing the protocol with OTS personnel, it was decided that some modifications might be worth investigating. The development of MRI's final protocol is discussed below.

GC Analysis Conditions

The procedure used by BMI was based on direct injection of the aqueous or methanolic sample onto a Chromosorb 101 column. A nitrogen sensitive thermionic detector was used to achieve the necessary selectivity for AA in the presence of hydrocarbons. Initially, the acrylamide analyses at MRI were performed on a Perkin-Elmer Model 3920 instrument equipped with a nitrogen/phosphorus selective flame ionization detector. A 5 ft x 1/8 in. OD glass column packed with 80/100 mesh Chromosorb 101 was used with a helium carrier flow rate of 20 ml/min. The operating conditions were: injection temperature, 220°C; column temperature, 220°C; transfer line/detector temperature, 250°C; hydrogen flow, 3 ml/min; air flow, 65 ml/min. At these conditions AA elutes in 3.8 min. The sensitivity of the detector was monitored periodically by measuring the bead current. A constant bead current of 30 picoamperes was maintained by adjustment of the bead heating current with a jet potential control setting of 3. However, because of difficulties in maintaining sensitivity levels on that system, it was decided to investigate a different detection system. A Varian 3700 gas chromatograph equipped with a Hall Electrolytic Conductivity Detector (Tracor, Inc.) in the nitrogen mode was investigated. A 6 ft x 2 mm ID glass column packed with 80/100 mesh Chromosorb 101 was used with a helium carrier flow rate of 30 ml/min. Other operating conditions were: injector temperature, 240°C; column temperature, 220°C; detector base temperature, 240°C; detector furnace temperature, 820°C; hydrogen reactor gas flow, 35 ml/min; 15% isopropanol/water electrolyte flow rate, 0.5 ml/min. Under these conditions, AA eluted in 5.1 min. A calibration curve using AA in methanol is shown in Figure 2. The sensitivity and stability of the Hall detector appear to be better than that of the nitrogen/phosphorus selective AFID.

This system was then used for most of the method development studies and all of the sample analyses. In operation, the solvent was vented for 3 to 4 min following sample injection.

Table 2. BMI'S SAMPLING AND ANALYSIS PROTOCOL[1/]

Media	Sampling technique	Sample treatment and preparation	Verification or recovery
Air			
Vapor	Midget impinger with 35 ml of 40% methanol-water. Sample 1-1.5 lpm for 24 hr. Store in a refrigerator.	Evaporate to 1 ml. Analyze as a water sample by direct injection.	None reported.
Particulates	Extract 3 times for 10 min with 7 ml methanol.	Evaporate to 0.5 ml or less. Analyze as a water sample by direct injection.	None reported.
Water	Grab sample of 1 liter.	Evaporate 20 ml to 0.5 ml or less by Kuderna Danish or slow nitrogen stream with heat. Analyze by direct injection.	10 ml of 1 ppm evaporated and reconstituted gave total recovery in silylated vessels.
Soil	Dry and extract a 50-g sample with 70 ml methanol. Repeat and filter the combined extracts.	Evaporate to 0.5 to 1 ml. Analyze as a water sample by direct injection.	None reported.

Air Samples

Recovery from Charcoal - The sampling method proposed but untested by BMI used water filled midget impingers that were preceded by a glass fiber filter. It was agreed at the project meeting that the impinger train had several disadvantages and that it would be worth investigating the use of solid adsorbents, e.g., charcoal, as the trapping medium. Accordingly, our work concentrated on evaluating several alternate air sampling trains.

In the first set of experiments, activated charcoal was tested as an adsorbent. The charcoal, Fisher Coconut charcoal, 8/15 mesh, was extracted with methanol and activated at 100 or 250°C under a flowing stream of nitrogen for 1 hr. A preliminary recovery experiment was made by packing two glass tubes, 8 mm OD x 6 mm ID x 60 mm length, with about 1 g of charcoal activated at 100 or 250°C. Acrylamide, 100 µl of 1 ppt in methanol, was injected onto the top of the two columns. The tubes were eluted directly with methanol and successive 4-ml aliquots were collected for analysis. In both cases, the AA that was desorbed completely eluted in the first 12 ml of methanol. The total recoveries were 57% and 30% for charcoal activated at 100 and 250°C, respectively. The results suggested that AA was being irreversibly adsorbed on highly reactive sites and that the high temperature charcoal had more reactive sites. A reverse desorption experiment was made by adding 5 ml of 10 ppm AA in methanol to 2 g of the 100°C charcoal. The solution level of AA was monitored periodically for 40 min. At that time, the "free" AA had dropped from 100 to 62%, indicating that the charcoal was actually adsorbing AA from the methanol. Thus, while charcoal might be a good trap for AA, the low recovery by methanol elution encouraged the search for another adsorbent.

Recovery from Tenax® and Chromosorb 101 - The same reverse desorption experiment was repeated using 80/100 mesh Chromosorb 101 and 60/80 mesh Tenax®-GC. After 40 min, the "free" AA was 93 and 98%, respectively. Another set of experiments with the glass tubes was run, using charcoal (100°C activation), Tenax® and Chromosorb 101. The tubes held about 1, 0.6, and 0.7 g of adsorbent, respectively. Each adsorbent was spiked with 100 µl of 1 ppt AA in methanol and air was drawn through at 0.5 liter/min for 30 min. The tubes were inverted and eluted with two 10-ml aliquots of methanol. The recoveries were 65, 110 and 90%, respectively, and all the AA was found in the first 10 ml of methanol.

A related experiment was then performed in an attempt to transfer the AA to the adsorbent as a vapor rather than a solution. Acrylamide was vaporized using the apparatus shown in Figure 1.

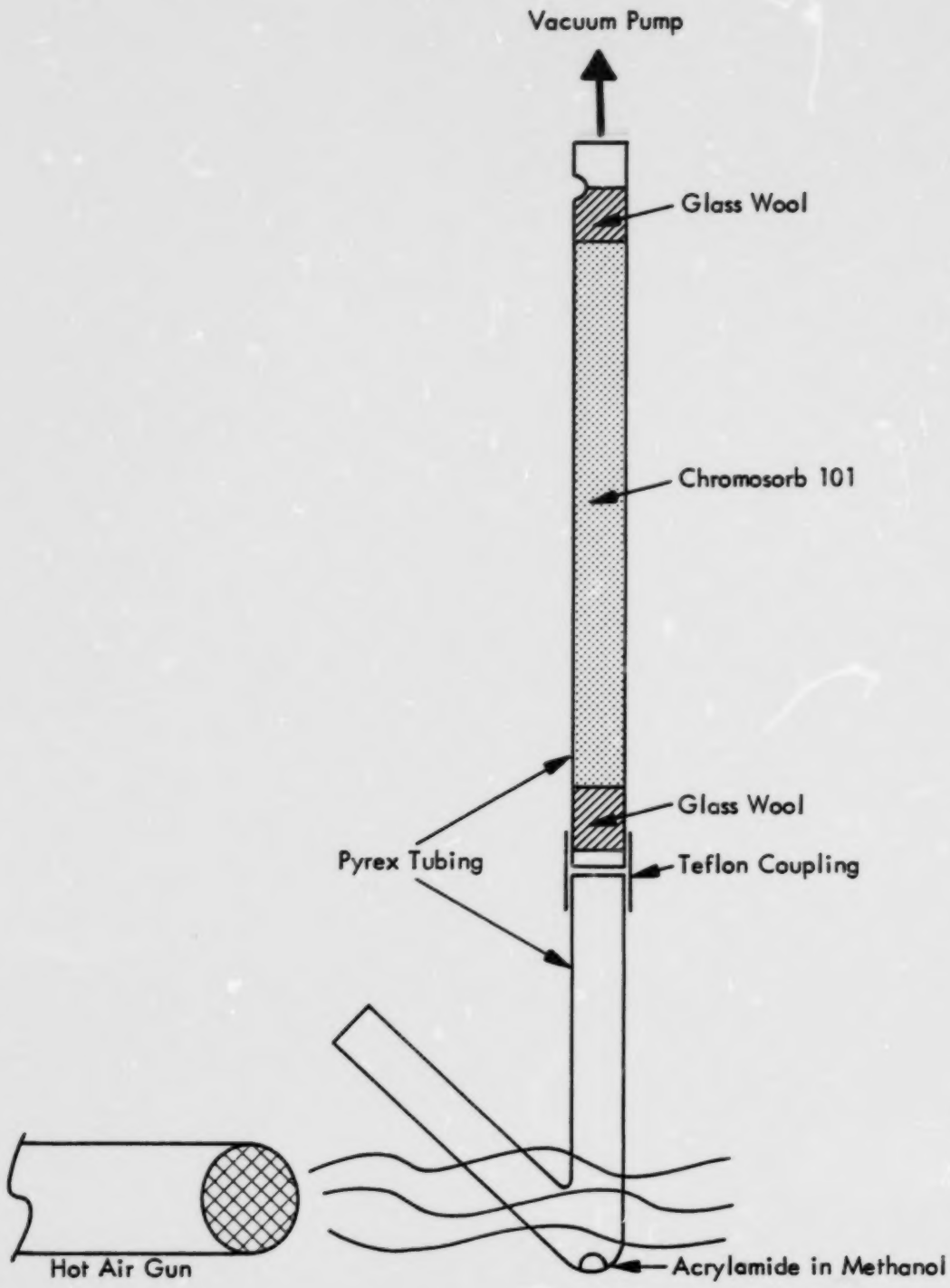


Figure 1. Vaporization of acrylamide for air sampling studies

Air was pulled through the system for 1 hr at 0.5 liter/min. The inside temperature of the U-tube was measured at ~ 100°C. Acrylamide was removed from the Chromosorb 101 by elution with 10 ml methanol and an 83% recovery was achieved. This level of recovery was felt to be within experimental error of the 90% recovery achieved following the direct application of the AA in methanol. Subsequent recovery experiments were done by direct injection of AA in methanol onto the adsorbent. Chromosorb 101 was tentatively chosen over Tenax® for the sampling train for reasons of availability and cost.

In the next experiment, Chromosorb 101 was spiked with 200 µg AA and air was drawn through the tube for 16 hr at 0.6 liters/min. After elution with 10 ml methanol, a 90% recovery of AA was found. Additional experiments run with longer sampling times and higher flow rates are discussed below.

Evaporation of Methanol Extracts - The size of the adsorbent traps being evaluated required 10 ml of methanol for quantitative elution. The possibility of reducing this volume to 1 ml or less by evaporation was investigated. The objective of the study was to lower the air level limit of detection by increasing the analyte concentration through evaporation. Four 25-ml aliquots of 10-ppm acrylamide in methanol were evaporated to 1 ml at 25 and 50°C in silylated and unsilylated vials, under a stream of nitrogen. The recoveries are given in Table 3.

Table 3. RECOVERY OF ACRYLAMIDE FOLLOWING EVAPORATION

Temperature	Silylated vial	Unsilylated vial
25°C	83%	74%
50°C	83%	83%

Thus solutions of AA in methanol can be effectively evaporated at 50°C in silylated or unsilylated glassware. As a precaution, however, silylated glassware was used routinely.

Recovery at Various Loading Levels - To determine the percent recovery at various levels of loading, 2.9 to 214 µg of AA was loaded by injection onto 0.7 g 60/80 mesh Chromosorb 101 packed into silanized glass tubes. The larger mesh adsorbed was used to reduce the pressure drop across the sampling tube. Each tube was connected to a small pump and air was drawn through the tube at a rate of 325 to 450 ml/min. After 24 hr, each tube was inverted and eluted with 10 ml methanol. The eluates from the tubes loaded with 100 to 200 µg AA were analyzed directly. The eluates from the tubes loaded with 2.9 to 29 µg AA were evaporated from 10 ml volume to approximately 1 ml using a 50°C water bath and a gentle stream of nitrogen. In order to calculate the exact volumes of the evaporated samples, a known

amount of benzonitrile in methanol was added to each. The samples were then analyzed and quantitated using a mixed AA-benzonitrile standard. The percent recovery for each sample was determined and is listed in Table 4. The data are also graphed in semi-log form in Figure 2. The four points were averaged to obtain a 99% recovery level.

Table 4. PERCENT RECOVERY OF ACRYLAMIDE

µg Acrylamide loaded	µg Acrylamide recovered	Percent recovery	Average
2.90	3.01	104	104%
29.0	38.7	133	
29.0	39.7	137	121%
29.0	26.6	92	
107	96.0	90	90%
214	156	73	
214	171	80	77%
214	168	79	

Recovery at Various Air Volumes - An experiment was then performed to determine the effect on recovery of the volume of air sampled in a 24-hr period. Four silanized glass tubes packed with 0.7 g Chromosorb 101 were loaded with 214 µg AA by injection of a methanolic standard. Each tube was connected to a small vacuum pump and air was drawn through at a rate of 250 to 600 ml/min. At the end of the 24-hr period, each tube was inverted and eluted with 10 ml methanol and analyzed. The percent recovery was determined and is listed in Table 5.

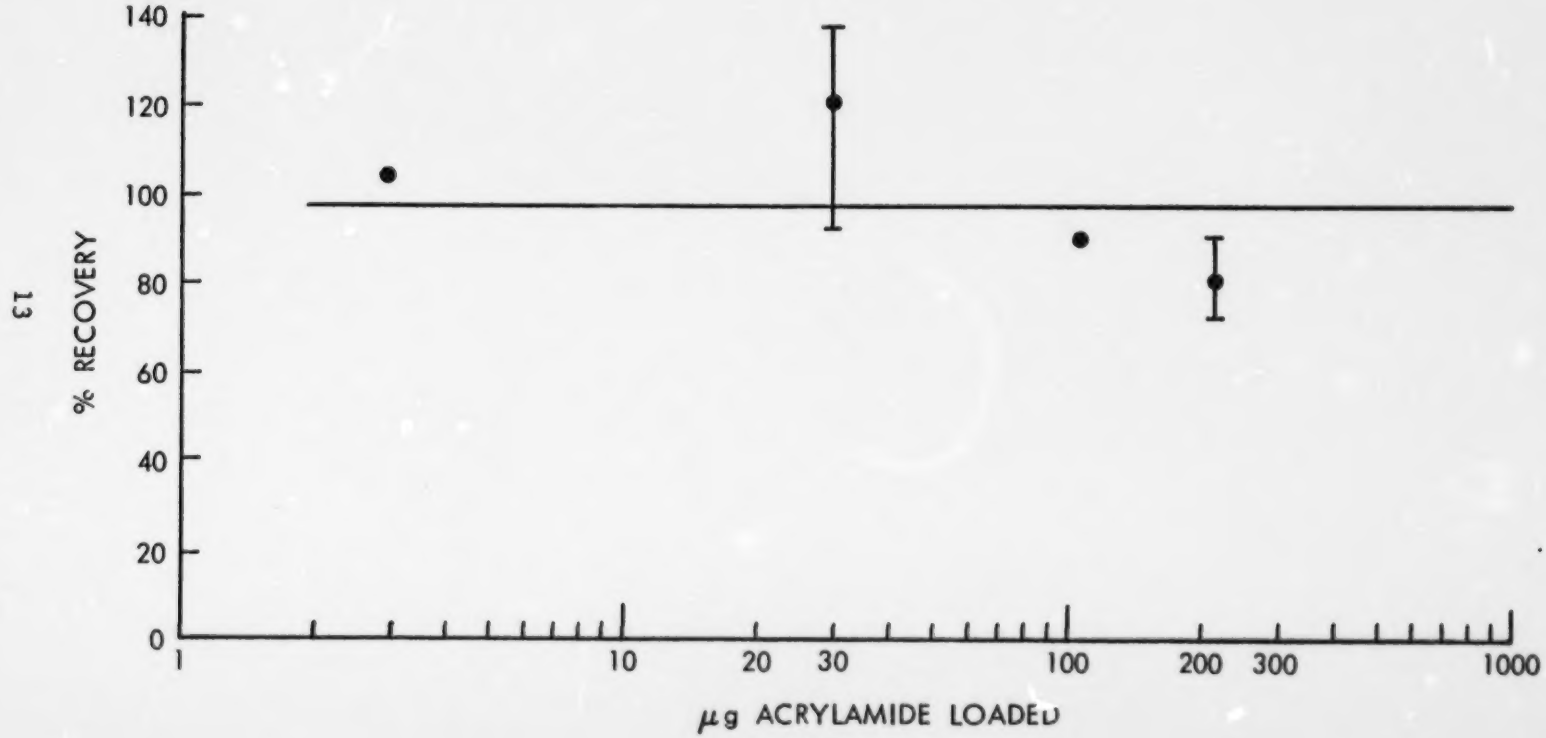


Figure 2. Recovery of acrylamide versus level of acrylamide

Table 5. PERCENT RECOVERY VERSUS VOLUME OF AIR

Sample	µg Acrylamide loaded	µg Acrylamide recovered	Air flow rate (ml/min)	Total air volume (ℓ)	Percent recovery
1	214	168	250	360	79
2	214	171	370	540	80
3	214	156	380	600	73
4	214	123	600	880	57

It appeared that the percent recovery was decreasing at the higher flow rate. It was concluded that air sampling should be done at 500 ml/ min or less for best recovery. If a high flow rate is desired, the use of a backup tube is mandatory.

Water Samples

Recovery from Charcoal - The procedure first proposed by BMI[1/] was based upon the adsorption of AA from water with a column of activated charcoal. It was reported that 1,000 ml of water, 1 ppm in AA, could be passed through 2 g of petroleum-based charcoal (30/60 mesh, SKC, Inc.) and the AA eluted with 20 ml methanol with a 76% recovery. A greater concentration factor could be achieved by evaporating the methanol to 1 ml or less.

The experiment was repeated at MRI with less success. Two grams of the Fisher charcoal activated at 100°C were packed into a column and 1,000 ml of water, 1.07 ppm in AA, was passed through at 10 ml/ min. The charcoal was dried with a nitrogen gas stream and eluted with 25 ml methanol. Only 16% of the expected AA was recovered. It was not established whether the low recovery was due to breakthrough or incomplete desorption with methanol.

Direct Evaporation - It was also reported by BMI[1/] that aqueous AA solutions could be preconcentrated by direct evaporation.

Several direct evaporation experiments were conducted using conventional rotary evaporator equipment. Increasing volumes of water with decreasing AA concentrations were evaporated with a 50°C heated bath, to less than 1 ml. At the higher levels, the concentrate was diluted to 10.0 ml and analyzed directly. At the lower concentrations, a known amount of benzonitrile was added to the 1 ml of concentrate as an internal standard. The percent recoveries of the low level samples are given in Table 6.

Table 6. RECOVERY OF ACRYLAMIDE IN WATER FOLLOWING DIRECT EVAPORATION

Original volume (ml)	Acrylamide (ppm)	Final volume	Percent recovery
100	1.00	10.0	92
100	0.100	2.2	74
1,000	0.010	0.89	71

It was concluded that direct evaporation could be used on the real samples with an acceptable recovery.

Soil Samples

Two 10-g samples of Kansas City garden soil were spiked with 107 µg of AA. The first was packed into a chromatography column and eluted with methanol. Five successive 5-ml aliquots were collected and analyzed. The first 5-ml fraction was found to contain all of the added AA. The second 10-g sample was covered with 10 ml methanol in a vial and sonicated for 5 min. The sample and methanol were then centrifuged and the extract removed and analyzed. The recovery of AA was 75%. An unspiked 10-g sample was also eluted with 5 ml methanol and found to contain no AA.

For analysis of field soil samples, an elution with methanol will be used.

Sediment Samples

The sediment sample collected from the Calgon site was used for the development of the sediment analysis protocol. Six sediment samples of approximately 10 g each were centrifuged and the supernatant water was removed. Four of the samples were spiked with 100 µg of AA. Two of the spiked samples were transferred to columns and eluted successively with three 5-ml aliquots of methanol. The total recoveries of AA from the samples were 83 and 65%.

The other two spiked samples were sonicated 1 hr with 10 ml methanol. In this case, 76 and 81% of the added AA were recovered. No AA was recovered from the unspiked sediment using either elution technique.

The sonication technique was used for the analysis of real samples due to its experimental simplicity and slightly better precision. Approximately 50 g of sediment was sonicated successively with two 50-ml aliquots of methanol. The methanol extracts were combined and evaporated to 5 ml prior to analysis.

SECTION IV

SELECTION OF SAMPLING SITES

A meeting was held at the Office of Toxic Substances, EPA, in June 1977 to discuss the selection of sampling sites. Present were Dr. J. Hillman and Dr. P. Hilgard of OTS, and Dr. J. Going from MRI.

Table 7 lists the production and user sites that had been proposed for sampling by BMI. Standard Oil of Ohio was eliminated as it no longer produced AA. American Cyanamid, New Orleans, was eliminated based on the remarks of Dr. Bikales, National Science Foundation, that the plant would be difficult to monitor because of its location. It was suggested that Du Pont probably uses AA as a thickening agent for gelled explosives.

Table 7 also lists the user sites proposed by BMI. Betz was established to be a minor user and Celanese no longer used AA. The paper mills were eliminated on the suggestion of Dr. Bikales.

With the assistance of Bob Peterson of MRI's North Star Division, additional information was gathered about the producers and users of AA. A new list of recommended sampling sites was developed and discussed with J. Hillman and P. Hilgard of OTS. This list, given as Table 8, differs in the following ways.

The Dow Chemical plant at Midland, Michigan, had been sampled by BMI and was eliminated. The Bio-Rad plant is a minor producer and user and was unlikely to have significant emissions of AA. The American Cyanamid plant at Linden, New Jersey, was not recommended for several reasons. The plant will switch to the catalytic conversion of acrylonitrile as the production process by 1978. The physical location of the plant made off-plant air sampling extremely difficult. Furthermore, the aqueous wastes are barged out into the Atlantic Ocean and dumped.

Table 7. BMI'S LIST OF ACRYLAMIDE PRODUCTION AND USER SITES[1/]

Acrylamide production sites	Recommended production sites
Dow Chemical, USA, Midland, Michigan	Dow Chemical, Midland, Michigan
DuPont, Belle, West Virginia	DuPont, Belle, West Virginia
Bio-Rad Laboratories, Richmond California	Bio-Rad Laboratories, Richmond, California
American Cyanamid, Linden, New Jersey	American Cyanamid, Linden, New Jersey
American Cyanamid, Fortier Louisiana	
Standard Oil of Ohio, Lima, Ohio	
Acrylamide user sites	**Recommended user sites**
Hercules, Inc., Hopewell, Virgina	Hercules, Inc., Hopewell, Virginia
Nalco Chemical Company, Chicago, Illinois	Nalco Chemical Company, Chicago, Illinois
Calgon Corporation, Pittsburgh, Pennsylvania	Calgon Corporation, Pittsburgh, Pennsylvania
Pascagoula, Mississippi (soil stabilization)	Soil stabilization site to be specified
Wenatchee, Washington (soil stabilization)	Soil stabilization site to be specified
Betz Laboratory, Trevase, Pennsylvania	
Celanese, Charlotte, North Carolina	
Kimberly-Clark, Appleton, Wisconsin	
Mead Paper, Chillicothe, Ohio	

Table 8. RECOMMENDED SAMPLING SITES

Site	Location	Category	Type of operation/process, comments
Monomer and polymer producers			
American Cyanamide	Fortier, Louisiana	Monomer producer Polymer producer	Catalytic conversion of acrylonitrile
Nalco	Garyville, Louisiana	Monomer producer Polymer producer	Catalytic conversion of acrylonitrile
Polymer producers			
Calgon	Pittsburgh, Pennsylvania	Polymer producer	
Hercules	Hopewell, Virginia	Polymer producer	
-	Unidentified	Soil stabilization	
Polymer users			
St. Regis	Sartell, Minnesota	Polymer user	Manufacture of paper

It was recommended that the American Cyanamid plant at Fortier, Louisiana, be sampled instead. It was representative of the most commonly used production process and was both a producer and a user. It was also recommended that the Nalco plant at Garyville, Louisiana, be sampled as a producer and a user. This plant was not on the BMI producers list. Among the polymer producers, Nalco at Garyville (as a monomer and polymer producer) rather than at Chicago, Illinois, was recommended.

A paper manufacturer, in the category of a polymer user, was added to the recommended list. The St. Regis plant at Sartell, Minnesota, would be monitored for aqueous discharges only.

The Hercules plant in Hopewell, Virginia, the Calgon plant in Pittsburg, Pennsylvania, as well as a soil stabilization site, were recommended by both MRI and BMI. In the course of attempting to locate a suitable soil stabilization site, MRI was advised by Mr. David Withers, Regional Manager, Mining and Industrial Chemicals, American Cyanamid, that he was not aware of any suitable ongoing projects.

The locations of the sampling sites are shown in Figure 3.

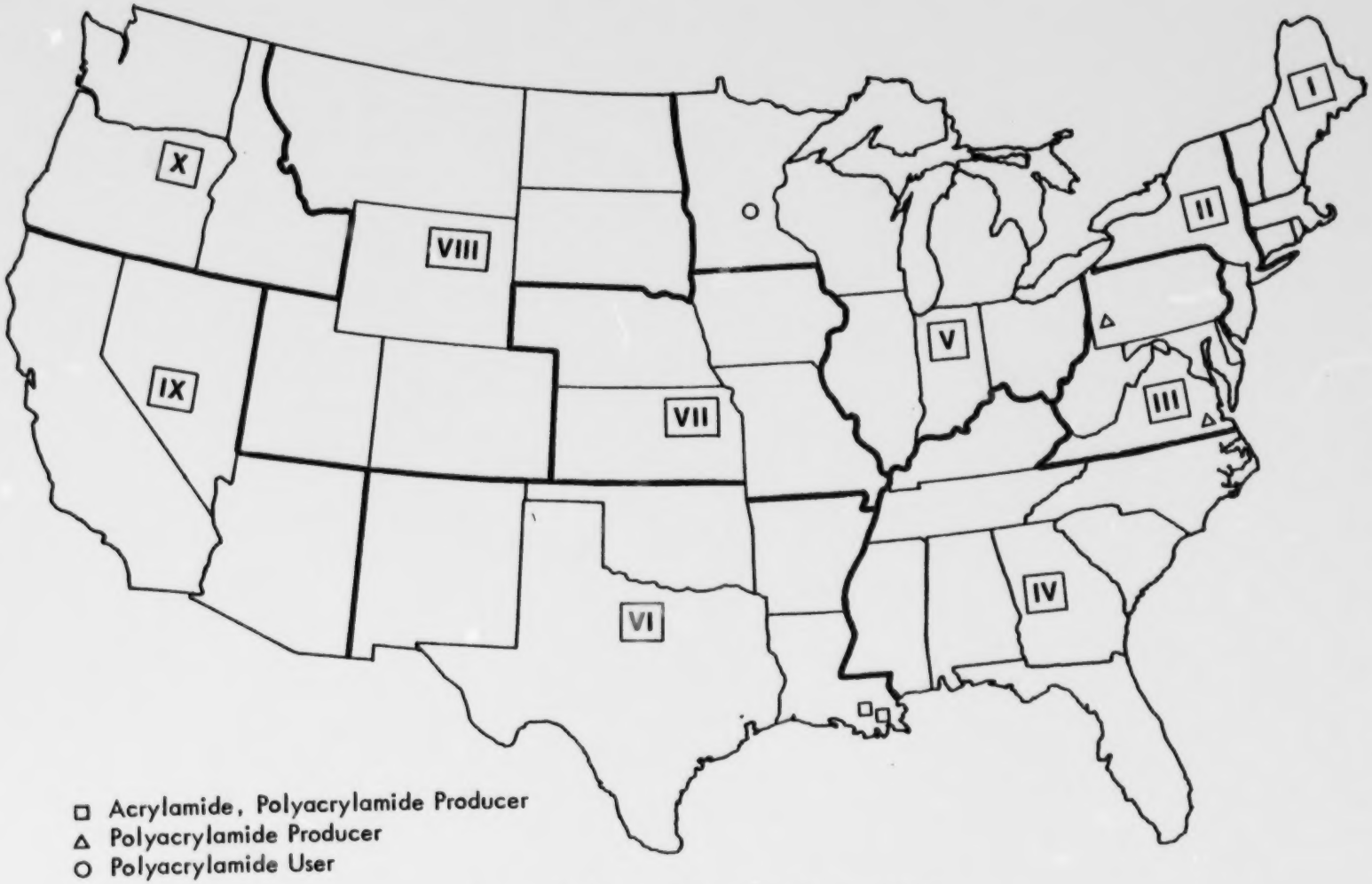


Figure 3. Location of sampling sites

SECTION V

DISCUSSION OF RESULTS

The five selected industrial sites were sampled between August 8, 1977, and October 6, 1977. The actual sampling schedule is shown in Table 9; presampling site visits were generally conducted on the day prior to sampling. A summary of the air and water samples collected at the five sites is given in Table 10 and a summary of the air sampling parameters follows in Table 11. The results obtained from the five industrial sites are discussed below. A sixth site was sampled previously by another contractor and these results are also given for comparison.

HERCULES, INC., HOPEWELL, VIRGINIA

Field Sampling

A presampling survey was conducted on August 8, 1977. The plant lies within the city limits of Hopewell, Virginia, south of the junction of state highways 10 and 156. Allied Chemical operates a large complex approximately 0.75 miles to the north-northeast of the Hercules plant. Approximately 0.75 miles to the northwest is a Firestone polymer plant. The municipal sewage treatment facility is located in the city landfill immediately south of the plant adjacent to Bailey Creek into which it discharges.

The NPDES permit application shows four discharge points, 001 through 004, which are labeled accordingly in Figure 4. All four flow into Bailey Creek which then discharges into the James River. These discharges receive some treatment before being discharged. At sampling time a regional sewage treatment facility was nearing completion at the junction of Bailey Creek and the James River. We were informed by Mr. Burnett of the State Water Control Board that the facility was being funded by the industrial sites, the City of Hopewell, and the U.S. Environmental Protection Agency and when completed would receive treated waste from the city and each industrial site. Discharge into Bailey Creek from Hercules would still occur during heavy rains.

Table 9. SAMPLING SCHEDULE

Date	Company	Location	Product or operation
8/8/77-8/10/77	Hercules, Inc.	Hopewell, Virginia	Polyacrylamide producer
8/30/77-8/31/77	Calgon Corporation	Ellwood City, Pennsylvania	Polyacrylamide producer
9/12/77-9/14/77	Nalco Chemical Company	Garyville, Louisiana	Acrylamide producer Polyacrylamide producer
9/14/77-9/16/77	American Cyanamid	Fortier, Louisiana	Acrylamide producer Polyacrylamide producer
10/6/77	St. Regis Paper Company	Sartell, Minnesota	Polyacrylamide user

Table 10. FIELD SAMPLING SUMMARY

Site	Air samples (stations x components)	Total samples (number/type)
Hercules	5 x 2	10 Air 4 Soil 4 Water 1 Sediment
Calgon	4 x 2	8 Air 4 Soil 4 Water 1 Sediment
Nalco	4 x 2	8 Air 3 Soil 3 Water
American Cyanamid	4 x 2	8 Air 4 Soil 3 Water 2 Sediment
St. Regis	-	2 Water
Dow Chemical[a/]	-	-

a/ Samples collected and analyzed by separate contractor. See Ref. 1.

Table 11. SUMMARY OF AIR SAMPLING PARAMETERS

Site	Average sample volume (ℓ) (no. of samples)	Average sampling time (hr)	Average sampling rate (ℓ/min)
Hercules	402 (5)	25.9	0.26
Calgon	1,008 (4)	25.5	0.66
Nalco	722 (4)	20.1	0.60
American Cyanamid	893 (4)	23.4	0.64
Dow Chemical[a/]	-	-	-

a/ Samples collected and analyzed by separate contractor. See Ref. 1.

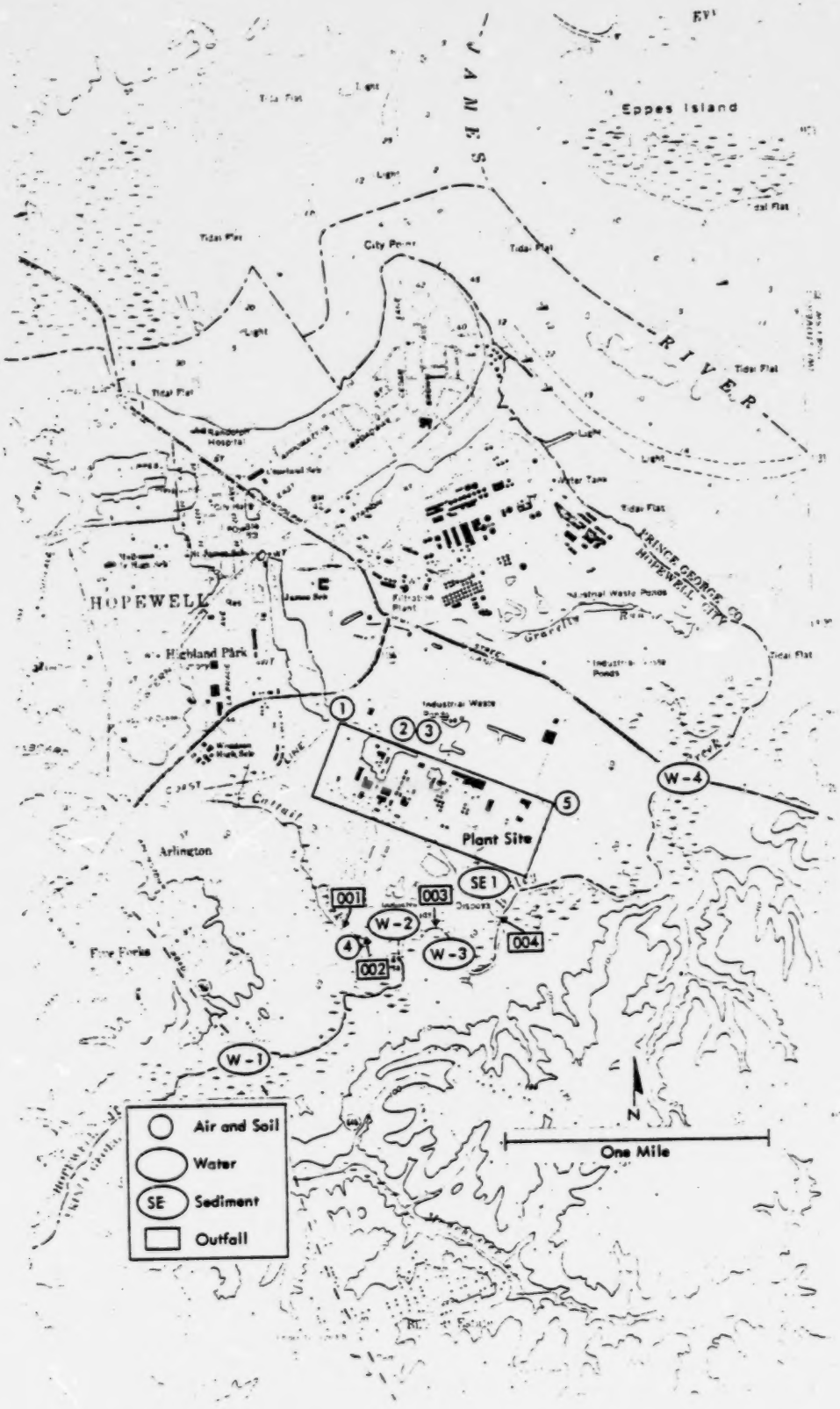


Figure 4. Sampling locations at Hercules, Inc., Hopewell, Virginia

The Hercules plant manufactures various celluloses, chlorine, chloroacetic acid, ethyl ether, hydrochloric acid, sodium sulfate, and polyacrylamide.

Local winds were reported to come primarily out of the southeast at that time of year.

Field sampling was carried out during the period from 0900 August 9, 1977, to 1300 August 10, 1977. Five air samplers were deployed at four sites upwind, downwind, and laterally with respect to the plant. Soil samples were taken from the air sampling sites, water samples were taken upstream and downstream from the point of discharge, and a sediment sample was taken below an outfall believed to be No. 004.

Air Sampling - Air sampling was conducted using five samplers at four sites. The sites were established upwind, downwind, and laterally with respect to the plant with two samplers located at the downwind site. Samplers were positioned at a height of 1.5 m on public land such as rights-of-way. Samplers were located at 0 and 1/8 mile from the plant perimeter. Locations of the five stations are shown in Figure 4. Site descriptions and air sampling data are given in Table 12.

Water Sampling - Four water samples were taken; two grab samples and two composite samples. The first composite sample, W-1, was taken over a 14-hr period from 1900 August 9, 1977, to 0900 August 10, 1977, from the bridge where Highway 156 passes over Bailey Creek upstream of the plant and municipal sewage treatment facility. The second composite sample, W-4, was taken over a 14-hr period from 2400 August 9, 1977, to 1400 August 10, 1977, from the bridge where Highway 10 passes over Bailey Creek downstream of the plant. Grab samples were taken at locations W-2 and W-3. The water sampling locations are shown in Figure 4 and described more thoroughly in Table 13.

Soil Sampling - Four soil samples were taken on August 10, 1977, one from each of the air sampling sites (see Table 12). Soil samples were taken by removing approximately the top 12 mm of soil from the area close to the air sampler.

Sediment Sampling - A sediment sample was taken from below an outfall believed to be No. 004 which, according to the NPDES permit application, should contain process waste from noncellulose polymer production. The sample was taken by scraping up some of the clay soil beneath the water level. The sampling data are given in Table 13.

Table 12. AIR SAMPLING DATA FOR HERCULES, INC., HOPEWELL, VIRGINIA

Area	Sample[a/] No.	Exact location, USGS coordinates		Total sampling (hr)	Sampling rate (ℓ/min)	Total volume (m^3)	Sample height (m)
Northwest transect, 0 km	A-1 (So-1)	0 km northwest at plant gate	4129450 m N 297560 m E	25.3	0.52	0.79	1.5
North transect, 0 km	A-2 (So-2)	0 km north on plant road	4129320 m N 298150 m E	26.9	0.29	0.46	1.5
North transect, 0 km	A-3 (So-3)	0 km north on plant road	4129320 m N 298150 m E	26.9	0.40	0.65	1.5
South transect, 0.3 km	A-4 (So-4)	0.3 km southwest at Hopewell sewage plant	4128120 m N 297310 m E	25.1	0.18	0.28	1.5
Northeast transect, 0 km	A-5 (So-5)	0 km east at northeast corner of plant	4128880 m N 298830 m E	25.1	0.62	0.93	1.5

a/ Soil samples were collected at these air sampling stations.

Table 13. WATER AND SEDIMENT SAMPLING DATA FOR HERCULES, INC., HOPEWELL, VIRGINIA

Sample No.	Exact location, USGS coordinates		Sample type	Volume collected
W-1	Highway 156 at Bailey Creek	4127320 m N 296950 m E	Composited water	1,000 ml
W-2	Bailey Creek below Outfall 002	4128140 m N 297330 m E	Grab water	1,000 ml
W-3	Bailey Creek below Outfall 003	4128050 m N 298240 m E	Grab water	1,000 ml
W-4	Highway 10 at Bailey Creek	4129050 m N 297810 m E	Composited water	1,000 ml
Se-1	Bailey Creek below Outfall 004	4128430 m N 298380 m E	Sediment	1,000 cm^3

Meteorological Conditions

The wind conditions existing at Byrd Airport, Richmond, Virginia, between 0800 August 9, 1977, and 1300 August 10, 1977, are summarized in Table 14. Locally winds blew predominantly from the southwest.

Sample Analysis

Air Samples - The results of the analysis of the five air samples and two blanks are listed in Table 15. No AA was found in the Chromosorb 101 extracts or in the filter extracts. The minimum detectable concentration, which was dependent upon the volume of air collected, ranged from 0.3 to 1 $\mu g/m^3$. The absence of AA in air sample No. 3 was later confirmed by GC/MS (vide infra).

Water Samples - The results of the analysis of the water samples are given in Table 16. No AA was found in Samples W-1, W-2, or W-3. The limit of detection for these samples, taking into account the 500-fold increase in concentration by evaporation, was approximately 0.8 μg/liter. Sample 4, however, was found to contain 1.5 ppm AA. This was the only sample taken downstream of outfall No. 004. The presence of AA in the sample was confirmed by GC/MS (vide infra).

Soil and Sediment Samples - The results of the analysis of the soil and sediment samples are listed in Table 17. No AA was found in any of the samples. The limit of detection was approximately 0.08 $\mu g/g$.

Confirmation of Identity by GC/MS - Confirmation of identity was made by GC/MS. It was originally planned to confirm AA by full scan or selected ion monitoring (SIM) GC/MS. The full mass spectrum of AA was obtained using a heated direct inlet and is shown in Figure 5. During the first GC/MS studies, a considerable amount of column bleed, particularly for m/e 71 and 55, was observed. Since these were the two most intense peaks for AA, the possibility of using either full scan or SIM GC/MS was virtually eliminated. Ultimately, high resolution SIM was used for confirmation. The operating conditions are listed below.

Instrument: Varian 311A Mass Spectrometer/Varian 2700 Gas Chromatograph
Data system: Varian Spectrosystem 100 MS
Separator: Watson-Biemann
Mass monitored: 71.0371
Resolution: ~ 7,000
Ionization potential: 70 ev
Emission current: 1 mA
Multiplier gain: $4 \times 10^6 - 2 \times 10^7$

Table 14. WEATHER CONDITIONS DURING SAMPLING AT HERCULES, INC. HOPEWELL, VIRGINIA: SOURCE OF DATA, BYRD AIRPORT, RICHMOND, VIRGINIA

Time (EDT)	Wind		Precipitation
	Direction	Speed (knots)	
August 9, 1977			
0800	W	5	None
0900	NW	3	None
1000	N	3	None
1100	NE	3	None
1200	NNW	6	None
1300	N	8	None
1400	NE	5	None
1500	E	4	None
1600	S	5	None
1700	SW	3	None
1800	S	3	None
1900	E	5	None
2000	ENE	5	None
2100	E	6	None
2200	E	7	None
2300	SE	5	None
2400	SE	5	None
August 10, 1977			
0100	ESE	4	None
0200	E	4	None
0300	SE	4	None
0400	SE	4	None
0500	SSW	3	None
0600	S	3	None
0700	S	3	None
0800	SW	5	None
0900	SW	5	None
1000	SW	6	None
1200	WSW	9	None
1300	W	9	None

Table 15. ACRYLAMIDE CONCENTRATIONS IN AIR SAMPLES FROM HERCULES, INC., HOPEWELL, VIRGINIA

Sample No.	Sampling period		Volume (cu m)	Type of sample	µg found	µg/m³
A-1	8/9/77	1049-	0.79	Air[a]	< 0.3	< 0.4[c]
	8/10/77	1207		Particulate[b]	< 0.8	< 1.1
A-2	8/9/77	0924-	0.46	Air	< 0.3	< 0.6
	8/10/77	1219		Particulate	< 0.8	< 1.6
A-3	8/9/77	0924-	0.65	Air	< 0.3	< 0.5
	8/10/77	1219		Particulate	< 0.8	< 1.3
A-4	8/9/77	1035-	0.28	Air	< 0.3	< 1.1
	8/10/77	1144		Particulate	< 0.8	< 0.8
Blank 1	-		-	Air	< 0.3	-
				Particulate	< 0.8	-
Blank 2	-		-	Air	< 0.3	-
				Particulate	< 0.8	-

a/ Chromosorb 101 extracts.
b/ Millipore filter extracts.
c/ Based on instrumental detection limit and volume of air sampled.

Table 16. ACRYLAMIDE CONCENTRATIONS IN WATER SAMPLES FROM HERCULES, INC., HOPEWELL, VIRGINIA

Sample No.	Aliquot volume (ml)	Final volume (ml)	µg found	µg/ℓ
W-1	250	0.5	< 0.2	< 0.8[a]
W-2	250	0.5	< 0.2	< 0.8
W-3	250	0.5	< 0.2	< 0.8
W-4	250	8.3	383	1,500

a/ Based upon instrument detection limit and sample size.

Table 17. ACRYLAMIDE CONCENTRATIONS IN SOIL AND SEDIMENT SAMPLES FROM HERCULES, INC., HOPEWELL, VIRGINIA

Sample No.	Sample size wet (g)	Final volume (ml)	µg found	µg/g
So-1	50	5	< 4	< 0.08[a]
So-2	50	5	< 4	< 0.08
So-4	50	5	< 4	< 0.08
So-5	50	5	< 4	< 0.08
Se-1	50	5	< 4	< 0.08

a/ Based upon instrumental detection limit and wet sample size.

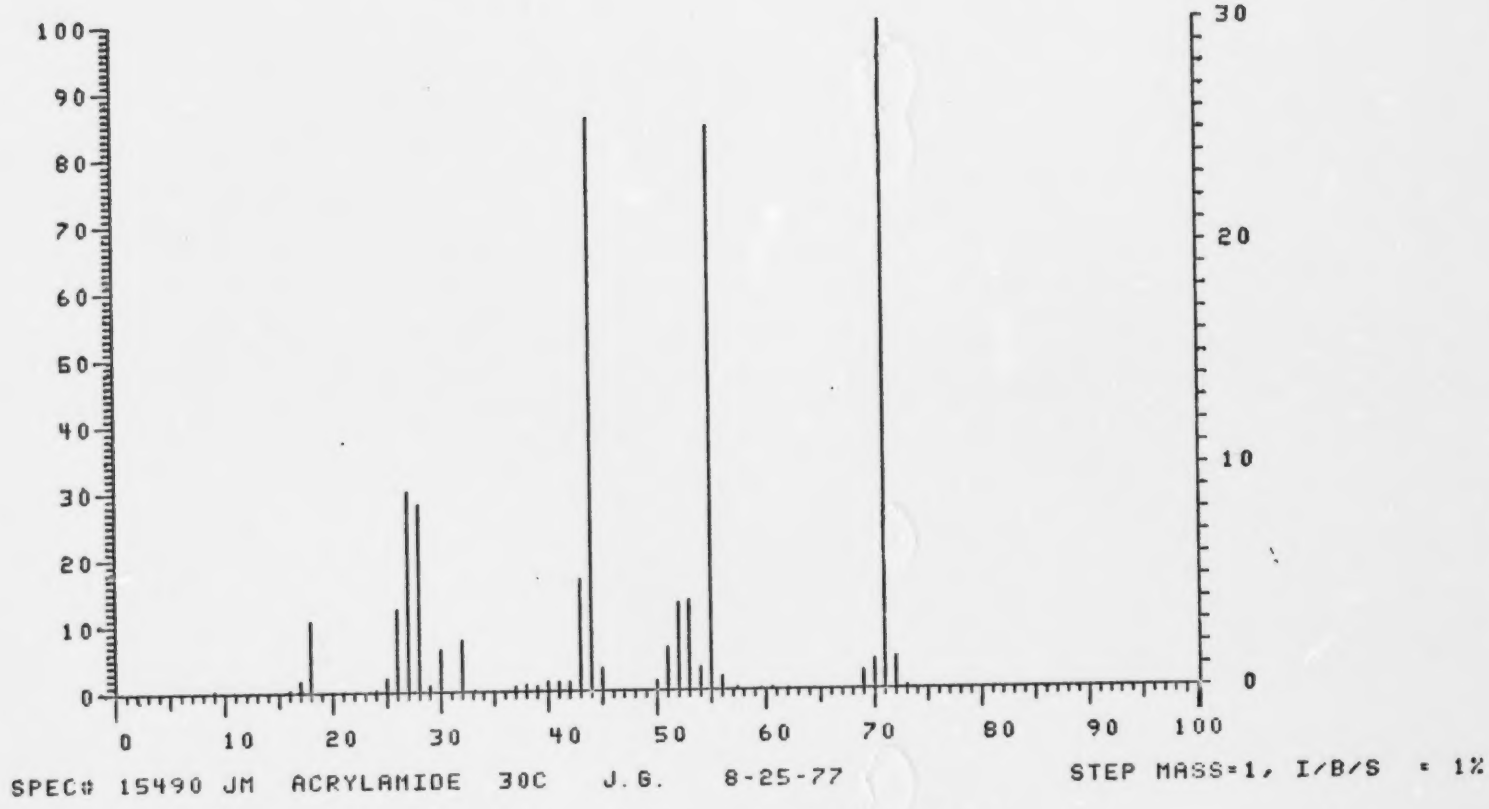


Figure 5. Mass spectrum of acrylamide

The resolution chosen, 7,000, was that necessary to provide baseline resolution of the AA 71 peak from the background 71 peak.

The presence of AA in water sample No. W-4 was first confirmed as shown by the ion current plots in Figure 6. The raw sample rather than the concentrated sample was analyzed. No attempt was made to quantitate the AA in W-4 by GC/MS.

The absence of AA in the concentrated air sample No. A-3 was also confirmed by this technique. The ion current plot is also shown in Figure 6 with the expected elution time of AA noted. The sensitivity of the GC/MS system in the high resolution mode was comparable to that of the GC/Hall detector. Thus, the limit of detection was about 0.5 $\mu g/m^3$ for air sample No. A-3.

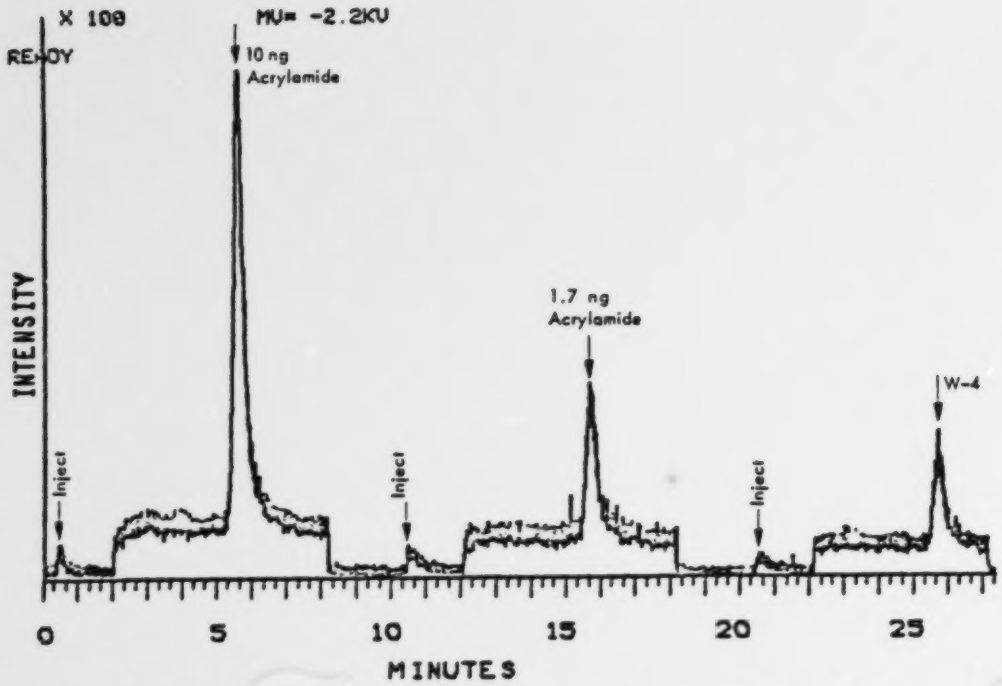
CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA

Field Sampling

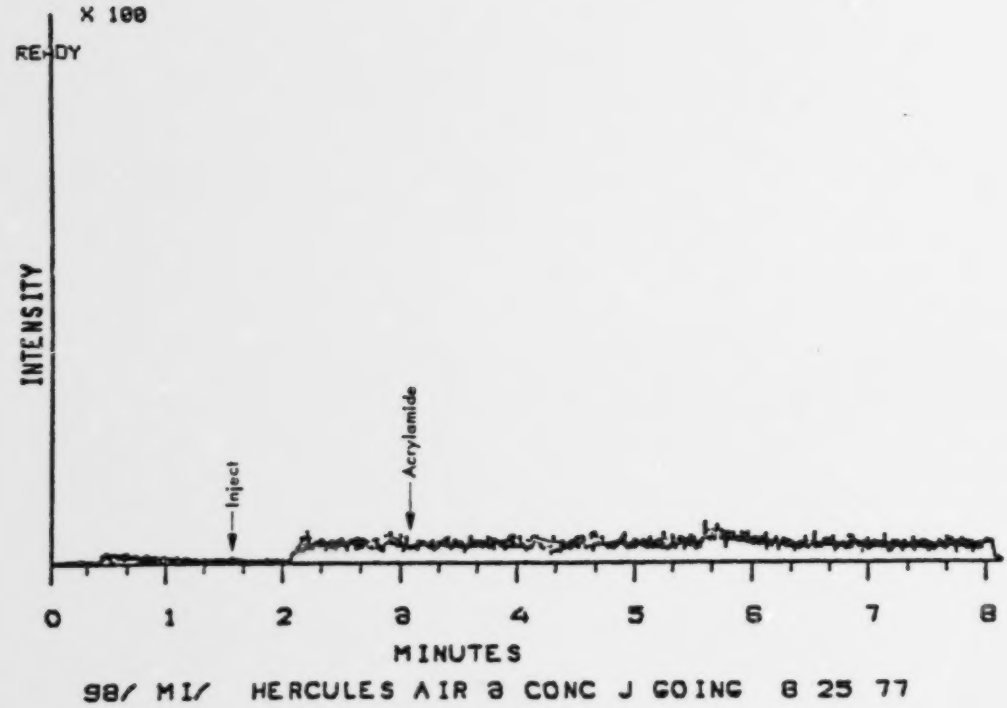
A presampling survey was conducted on July 25, 1977. The Calgon plant lies in Frisco Township of Beaver County, Pennsylvania, just east-southeast of Ellwood City, Pennsylvania. The plant is located in a semi-rural area near a bend in the Connoquenessing Creek, which lies to the north and west of the plant. The east side of the plant lies near an abandoned roadbed. There is a small chemical company adjacent to the plant on the south side which reportedly supplies Calgon with chlorine for water treatment.

The NPDES permit for this plant indicates that there are four discharge points to an unnamed tributary of Connoquenessing Creek. The tributary was found 200 yards east of a railroad trestle over River Road. This point is marked W-2 on the map shown in Figure 7. It was assumed that process water came from the local supply as no other source was located. This Calgon facility manufactures slowly soluble phosphates, water soluble polymers, and water treatment compounds.

Field sampling was carried out from 0800 (EDT) August 30, 1977, to 1300 (EDT) August 31, 1977. Four air samplers were deployed at four sites upwind, downwind, and laterally with respect to the plant. Soil samples were taken from each air sampling site; water samples were taken at various locations upstream and downstream from the points of discharge; sediment samples were taken below two discharge points.



a. Water sample W-4



b. Air sample A-3

Figure 6. Selected ion current plots, m/e = 71.0371

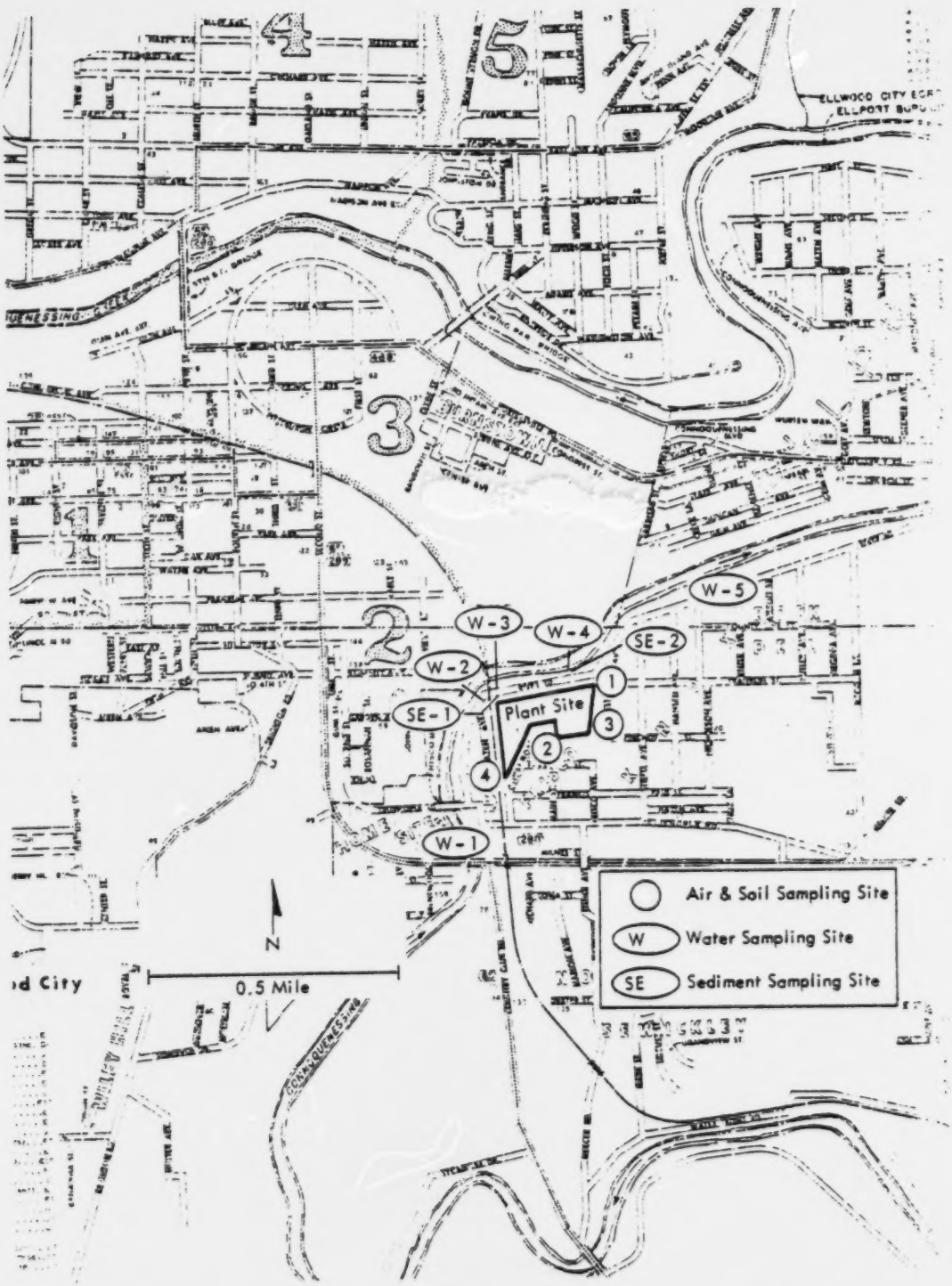


Figure 7. Sampling locations at Calgon Corporation, Ellwood City, Pennsylvania

Air Sampling - Air sampling was carried out from 0800 (EDT) August 30, 1977, to 1100 (EDT) August 31, 1977, using four samplers at four sites. The sites were located upwind, downwind, and laterally with respect to the plant. Samplers were positioned at a height of 1.5 m from the ground on public land such as rights-of-way. Locations of the samplers are shown in Figure 7. Site descriptions and air sampling data are given in Table 18.

Water Sampling - Five water samples were taken at various points upstream and downstream of the discharge points. All were composite samples taken during the period 0800 (EDT) August 30, 1977, to 1300 (EDT) August 31, 1977. Although the NPDES permit describes four discharge points into an unnamed tributary of the Connoquenessing Creek, only two outfalls were observed from the edge of the creek. One, W-4, is known to come from the plant and the other, W-3, may come from the plant. Both outfalls were large diameter concrete pipes which passed under River Road and flowed down the rocky bank of the Connoquenessing. Samples W-3 and W-4 were taken directly from outfalls; the remaining samples were taken from Connoquenessing Creek. Sampling locations are shown in Figure 7 and described in Table 19.

Soil Sampling - Soil samples were taken on August 31, 1977, at each of the air sampling sites (see Table 18) by scraping up the top 10 mm of soil.

Sediment Sampling - Sediment samples Se-3 and Se-4 were taken with water samples W-3 and W-4, which are at plant outfalls. Samples were taken by scraping some sediment up into jars. Sampling data are given in Table 19.

Meteorological Conditions

The wind conditions existing at Pittsburgh International Airport between 0800 (EDT) August 30, 1977, and 1100 (EDT) August 31, 1977, are summarized in Table 20. Locally the winds appeared calm during the period.

Sample Analysis

Air Samples - The results of the analysis of the four air samples are listed in Table 21. No AA was found in the Chromosorb 101 extracts or filter extracts. The limits of detection, which are dependent upon the volume of air actually sampled, were about 0.1 $\mu g/m^3$ for air and 0.4 $\mu g/m^3$ for particulates. No AA was found in the field blanks.

Table 18. AIR SAMPLING DATA FOR CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA

Area	Sample[a] No.	Exact location, USGS coordinates		Total sampling (hr)	Sampling rate (ℓ/min)	Total volume (m^3)	Sample height (m)
North transect, 0 km	A-1 (So-1)	0 km north on River Road	4522420 m N 561320 m E	25.6	0.80	1.22	1.5
South transect, 0 km	A-2 (So-2)	0 km south on Main Street	4522280 m N 561210 m E	25.5	0.63	0.97	1.5
East transect, 0 km	A-3 (So-3)	0 km east on Forest Grove Avenue	4522360 m N 561330 m E	25.5	0.58	0.89	1.5
West transect, 0 km	A-4 (So-4)	0 km west on Hazen Avenue	4522160 m N 560920 m E	25.5	0.61	0.95	1.5

a/ Soil samples were collected at these air sampling stations.

Table 19. WATER AND SEDIMENT SAMPLING DATA FOR CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA

Sample No.	Exact location, USGS coordinates		Sample type	Volume collected
W-1	0.5 km upstream of Outfall 001	4521950 m N 560930 m E	Composite water	1,000 ml
W-2	West side of railroad trestle	4522390 m N 560950 m E	Composite water	1,000 ml
W-3 Se-3	East side of railroad trestle, directly from Outfall 001	4522380 m N 561010 m E	Composite water Sediment	1,000 ml 1,000 cm^3
W-4 Se-4	0.3 km east of railroad trestle, directly from Outfall 002	4522480 m N 561300 m E	Composite water Sediment	1,000 ml 1,000 cm^3
W-5	0.6 km east of railroad trestle	4522750 m N 561720 m E	Composite water	1,000 ml

Table 20. WEATHER CONDITIONS DURING SAMPLING AT CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA: SOURCE OF DATA, PITTSBURGH INTERNATIONAL AIRPORT, PITTSBURGH, PENNSYLVANIA

Time (EDT)	Wind		Precipitation
	Direction	Speed (knots)	
August 30, 1977			
0800	WNW	3	None
0900	N	5	None
1000	WSW	3	None
1100	NE	4	None
1200	N	4	None
1300	NNE	4	None
1400	N	4	None
1500	-	0	None
1600	NW	3	None
1700	N	6	None
1800	N	5	None
1900	E	4	None
2000	SE	3	None
2100	E	3	None
2200	-	0	None
2300	-	0	None
2400	S	3	None
August 31, 1977			
0100	-	0	None
0200	-	0	None
0300	-	0	None
0400	-	0	None
0500	-	0	None
0600	-	0	None
0700	-	0	None
0800	SE	3	None
0900	-	0	None
1000	SE	3	None
1100	S	3	None

Table 21. ACRYLAMIDE CONCENTRATIONS IN AIR SAMPLES FROM CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA

Sample No.	Sampling period (hr)		Volume (m^3)	Type of sample	µg found	µg/m^3
A-1	8/30/77	0845-	1.22	Air[a]	< 0.1	< 0.1[c]
	8/31/77	1030		Particulate[b]	< 0.4	< 0.4[c]
A-2	8/30/77	0823-	0.97	Air[a]	< 0.1	< 0.1
	8/31/77	0945		Particulate[b]	< 0.4	< 0.4
A-3	8/30/77	0843-	0.89	Air[a]	< 0.1	< 0.1
	8/31/77	1014		Particulate[b]	< 0.4	< 0.4
A-4	8/30/77	0909-	0.95	Air[a]	< 0.1	< 0.1
	8/31/77	1040		Particulate[b]	< 0.4	< 0.4

a/ Chromosorb 101 extracts.
b/ Millipore filter extracts.
c/ Based upon instrument detection limit and sample size.

Water Samples - The results of the analysis of the five water samples are listed in Table 22. No AA was found in any of the samples after evaporation of 250 ml to 1 ml. The limit of detection was 0.8 µg/liter.

Soil and Sediment Samples - The results of the analysis of the soil and sediment samples are summarized in Table 23. No AA was found above the detection limit of 0.02 µg/g.

NALCO CHEMICAL COMPANY, GARYVILLE, LOUISIANA

Field Sampling

A presampling survey of the Nalco Chemical Company, Garyville, Louisiana, was conducted on September 12, 1977. The plant lies in St. John the Baptist Parish and is located on the north side of State Highway 44 about 2.25 miles northwest of Garyville, Louisiana. The plant lies in a rural area with the community of Mt. Airy located about 300 m to the northwest. Wastewater is discharged into the Mississippi River at the location shown in Figure 8. This plant produces AA and polyacrylamide through catalytic conversion of acrylonitrile.

Field sampling was conducted during the period 0900 September 13, 1977, to 1900 September 14, 1977. Four air samples and three soil samples were collected at that time. Water samples were collected later.

Air Sampling - Air sampling was conducted using four samplers at four sampling sites which were positioned upwind, downwind, and laterally with respect to the plant. Samplers were located at 0 and 1/8 mile from the plant property and were positioned at a height of 1.5 m on adjacent property, a railroad right-of-way, and a levee.

Sampling was carried out during the period 0900 (CDT) September 13, 1977, to 1900 (CDT) September 14, 1977, and was often interrupted by rain. The four sampling locations are shown in Figure 8. Site descriptions and air sampling data are given in Table 24.

Water and Sediment Sampling - Due to the extreme difficulty in obtaining a boat and the inclement weather, no water or sediment samples were taken by MRI personnel. Arrangements were then made with the Jefferson Parish Sheriff's Office to have one of their personnel collect water samples; these samples were received and analyzed. This office was involved in the collecting of a water sample from the American Cyanamid site at Fortier. For this reason, was is felt that samples of acceptable quality were collected. Sampling data are given in Table 25.

Table 22. ACRYLAMIDE CONCENTRATIONS IN WATER SAMPLES FROM CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA

Sample No.	Aliquot volume (ml)	Final volume (ml)	µg found	µg/ℓ[a]
W-1	250	1	< 0.2	< 0.8
W-2	250	1	< 0.2	< 0.8
W-3	250	1	< 0.2	< 0.8
W-4	250	1	< 0.2	< 0.8
W-5	250	1	< 0.2	< 0.8

a/ Based upon instrumental detection limit and sample size.

Table 23. ACRYLAMIDE CONCENTRATIONS IN SOIL AND SEDIMENT SAMPLES FROM CALGON CORPORATION, ELLWOOD CITY, PENNSYLVANIA

Sample No.	Sample size wet (g)	Final volume (ml)	µg found	µg/g
So-1	50	5	< 1	< 0.02[a]
So-2	50	5	< 1	< 0.02[a]
So-3	50	5	< 1	< 0.02[a]
So-4	50	5	< 1	< 0.02[a]
Se-1	50	0.5	< 1	< 0.02[a]
Se-2	50	0.5	< 1	< 0.02[a]

a/ Based upon instrument detection limit and wet sample size.

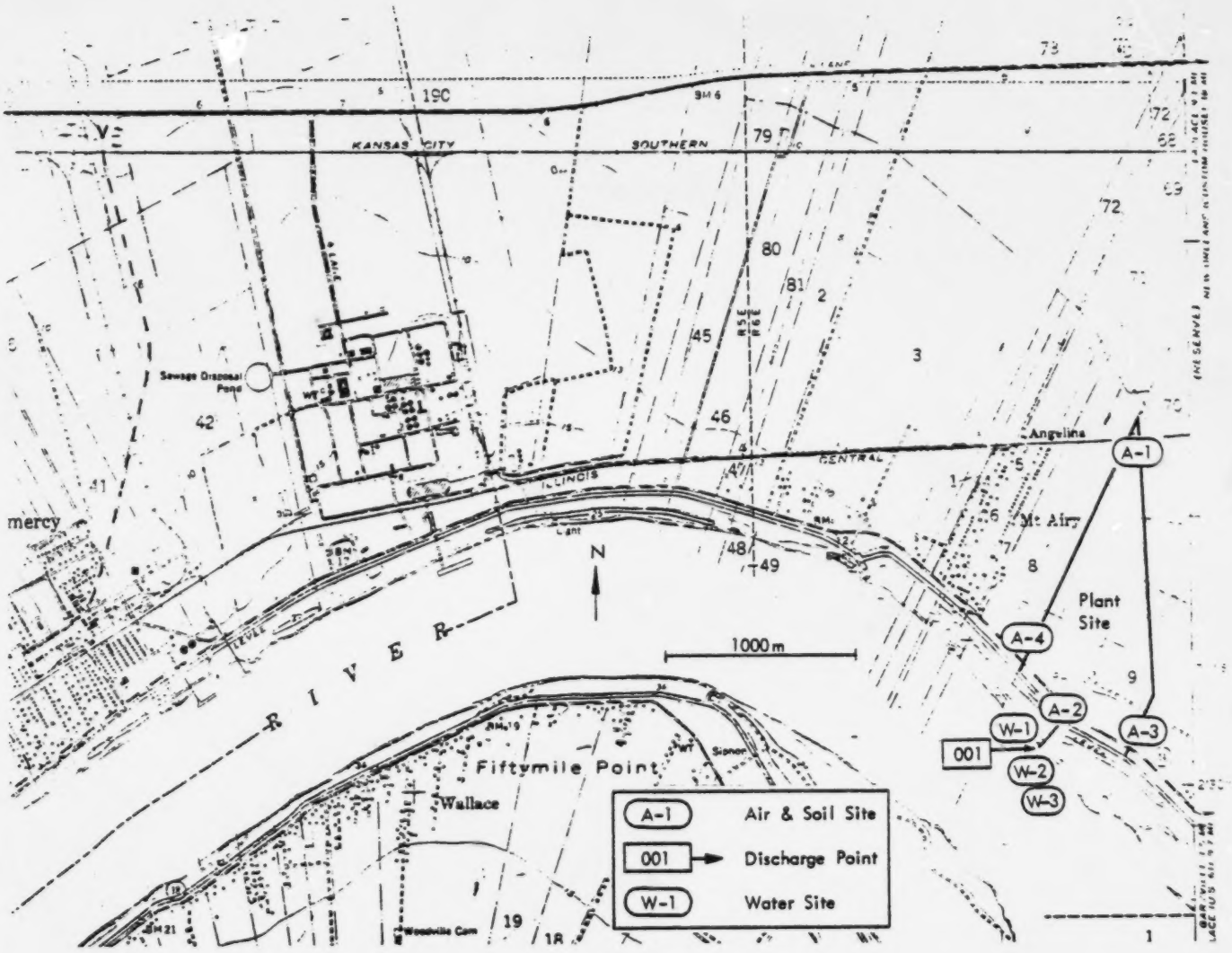


Figure 8. Sampling locations at Nalco Chemical Company, Garyville, Louisiana

Table 24. AIR SAMPLING DATA FOR NALCO CHEMICAL COMPANY, GARYVILLE, LOUISIANA

Area	Sample No.	Exact location, USGS coordinates		Total sampling (hr)	Sampling rate (ℓ/min)	Total volume (m^3)	Sample height (m)
North transect, 0 km	A-1	Plant spur and main line Illinois Central Railroad	3327400 m N 728720 m E	24	0.65	0.93	1.5
South transect, 0.2 km	A-2	0.2 km south on levee	3325960 m N 728300 m E	23.1	0.49	0.68	1.5
Southeast transect, 0 km	A-3	0 km east along State Highway 44	3325810 m N 728710 m E	10.6	0.63	0.40	1.5
West transect, 0 km	A-4	0 km west along fenceline	3326320 m N 728160 m E	23.5	0.62	0.88	1.5

Table 25. WATER SAMPLING DATA FOR NALCO CHEMICAL COMPANY GARYVILLE, LOUISIANA

Sample No.	Exact location, USGS coordinates		Sample type	Volume collected
W-1	30 m upstream of Discharge 001, 20 m offshore	3325820 m N 728270 m E	Grab	1,000 ml
W-2	At Discharge 001	3325770 m N 728290 m E	Grab	1,000 ml
W-3	150 m downstream of Discharge 001, 20 m offshore	3325650 m N 728390 m E	Grab	1,000 ml

Soil Samples - Soil samples were taken September 14, 1977, at three of the four air sampling sites by removing the top approximately 10 mm of topsoil from the area by each sampler. Sampling data are given in Table 26.

Meteorological Conditions - The meteorological conditions that existed during the sampling period were obtained from the National Weather Service, New Orleans International Airport, and are summarized in Table 27.

Sample Analysis

Air Samples - The results of the analysis of the four air samples and the blank are listed in Table 28. No AA was found in the Chromosorb 101 extracts or filter extracts. The limits of detection were about 0.15 $\mu g/m^3$ for air and 2 $\mu g/m^3$ for particulates. This level was established using spiked samples. No AA was found in the field blanks.

Water Samples - The results of the analysis of the three water samples are listed in Table 29. No AA was found above a limit of detection of about 0.8 µg/liter, established with spiked samples.

Soil Samples - The results of the analysis of the three soil samples are summarized in Table 30. No AA was present above the detection limit of 0.02 µg/g, established with spiked samples.

AMERICAN CYANAMID, FORTIER, LOUISIANA

Field Sampling

A presampling survey of the American Cyanamid Company plant, Fortier, Louisiana, was conducted on September 14, 1977. The plant is located in Jefferson Parish, Louisiana, in South Kenner along State Highway 18 about 6 miles east of Luling, Louisiana. The plant lies in a semi-rural area with the Mississippi River to the north. The NPDES permit shows two outfalls which discharge wastewater into the Mississippi River. Both are readily visible passing over the highway. The locations are shown in Figure 9. This plant produces AA and polyacrylamide through catalytic conversion of acrylonitrile.

Field sampling was conducted during the period 2000 September 14, 1977, to 0700 September 16, 1977. Four air samples, three water samples, four soil samples, and two sediment samples were collected.

Table 26. SOIL SAMPLING DATA FOR NALCO CHEMICAL COMPANY GARYVILLE, LOUISIANA

Sample No.	Exact location, USGS coordinates		Sample size	Percent moisture
So-1	Plant spur and main line Illinois Central Railroad	3327400 m N 728720 m E	250 cm^3	89
So-2	0.2 km south on levee	3325960 m N 728300 m E	250 cm^3	64
So-4	0 km west along fenceline	3326320 m N 728160 m E	250 cm^3	75

Table 27. WEATHER CONDITIONS DURING SAMPLING AT NALCO CHEMICAL, GARYVILLE, LOUISIANA: SOURCE OF DATA, NEW ORLEANS INTERNATIONAL AIRPORT, NEW ORLEANS, LOUISIANA

Time (CDT)	Wind		Precipitation[a/]
	Direction	Speed (knots)	
September 13, 1977			
0900	S	6	None
1000	S	9	None
1100	S	9	None
1200	S	3	None
1300	S	10	None
1400	SSW	11	None
1500	S	11	None
1600	S	8	Rain
1700	0	0	Rain
1800	NNE	3	Rain
1900	0	0	None
2000	NW	4	None
2100	E	3	None
2200	E	4	None
2300	ESE	4	None
2400	ESE	2	None
September 14, 1977			
0100	ESE	5	None
0200	SSE	4	Rain
0300	S	7	None
0400	S	5	None
0500	SSW	8	None
0600	NW	12	Rain
0700	W	8	None
0800	E	2	None
0900	SSW	7	Rain
1000	SSE	11	None
1100	S	10	None
1200	S	6	None
1300	SW	7	None
1400	S	10	None
1500	S	9	None
1600	S	7	None
1700	S	6	None
1800	SSE	7	None
1900	S	5	None

a/ Precipitation data from sampling site.

Table 28. ACRYLAMIDE CONCENTRATIONS IN AIR SAMPLES FROM NALCO CHEMICAL, GARYVILLE, LOUISIANA

Sample No.	Sampling period (hr)		Volume (m^3)	Type of sample	µg found	µg/m^3
A-1	9/13/77	0930-	0.93	Air[a]	< 0.1	< 0.11[c]
	9/14/77	1714		Particulate[b]	< 0.4	< 0.43[c]
A-2	9/13/77	1010-	0.68	Air[a]	< 0.1	< 0.15
	9/14/77	1806		Particulate[b]	< 3.2	< 4.7
A-3	9/13/77	1037-	0.40	Air[a]	< 0.1	< 0.25
	9/14/77	1045		Particulate[b]	< 1.6	< 4.0
A-4	9/13/77	1111-	0.88	Air[a]	< 0.1	< 0.12
	9/14/77	1740		Particulate[b]	< 1.6	< 1.8

a/ Chromosorb 101 extract.
b/ Millipore filter extract.
c/ Based upon instrument detection limit and sample size.

Table 29. ACRYLAMIDE CONCENTRATIONS IN WATER SAMPLES FROM NALCO CHEMICAL COMPANY, GARYVILLE, LOUISIANA

Sample No.	Aliquot volume (ml)	Final volume (ml)	µg found	µg/ℓ
W-1	250	1	< 0.2	< 0.8[a/]
W-2	250	1	< 0.2	< 0.8
W-3	250	1	< 0.2	< 0.8

a/ Based upon instrument detection limit and sample size.

Table 30. ACRYLAMIDE CONCENTRATIONS IN SOIL SAMPLES FROM NALCO CHEMICAL COMPANY, GARYVILLE, LOUISIANA

Sample No.	Sample size wet (g)	Final volume (ml)	µg found	µg/g
So-1	50	1	< 1	< 0.02[a/]
So-2	50	1	< 1	< 0.02
So-3	50	1	< 1	< 0.02

a/ Based upon instrumental detection limit and wet sample size.

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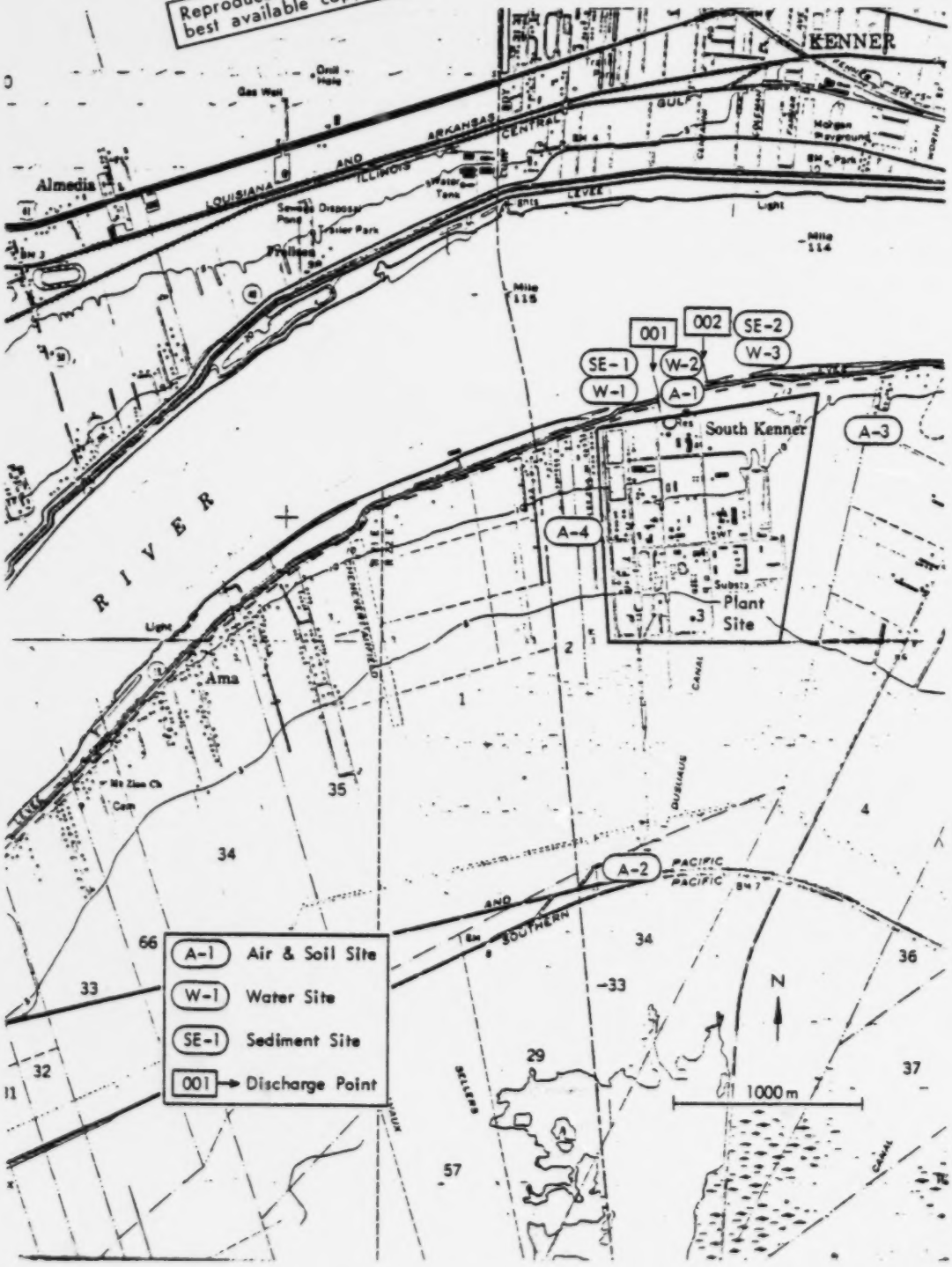

Figure 9. Sampling locations at American Cyanamid, Fortier, Louisiana

Air Sampling - Air sampling was carried out from 2000 (CDT) September 14, 1977, to 0700 (CDT) September 16, 1977, at four sampling sites located upwind, downwind, and laterally with respect to the plant. Samplers were located at 1/8 and 3/4 mile from the plant perimeter and were positioned at a height of 1.5 m on street rights-of-way, a railroad rightof-way, and on a levee. Sampling was interrupted by rain. The four sampling locations are shown in Figure 9. Site descriptions and air sampling data are given in Table 31.

Water and Sediment Sampling - Water and sediment samples were collected on September 16, 1977, from upstream, downstream, and between the two discharge points shown in Figure 9. Sampling data are given in Table 32.

Soil Sampling - Soil samples were taken on September 16, 1977, at each of the four air sampling sites by removing approximately the top 10 mm of soil from the area near each sampler. Sampling data are given in Table 33.

Meteorological Conditions - The meteorological conditions that existed during the sampling period were obtained from the National Weather Service, New Orleans International Airport, and are summarized in Table 34.

Sample Analysis

Air Samples - The results of the analysis of the four air samples and the blank are listed in Table 35. No AA was found in the Chromosorb 101 extracts or filter extracts. The limits of detection of about 0.12 $\mu g/m^3$ for air and 0.8 $\mu g/m^3$ for particulates were established using spiked samples. No AA was found in the field blanks.

Water Samples - The results of the analysis of the three water samples are given in Table 36. No AA was found above the detection limit of 0.8 μg/liter established using spiked samples.

Soil and Sediment Samples - The results of the analysis of the six soil and sediment samples are summarized in Table 37. No AA was present above 0.02 $\mu g/g$ as established using spiked samples.

ST. REGIS PAPER COMPANY, SARTELL, MINNESOTA

Field Sampling

A presampling survey of the St. Regis Paper Company plant in Sartell, Minnesota, was conducted on October 6, 1977. The plant is located in Benton County, Minnesota, just east of the corporate limits of Sartell, Minnesota. The plant is bounded on the west by the Mississippi River and on the east by State Highway 152. The DeZurik Company plant is located on the opposite side of the Mississippi River. Wastewater is discharged into the Mississippi River at the points shown in Figure 10. Only water and sediment samples were scheduled to be taken at this site.

Table 31. AIR SAMPLING DATA FOR AMERICAN CYANAMID, NEW ORLEANS, LOUISIANA

Area	Sample No.	Exact location, USGS coordinates		Total sampling (hr)	Sampling rate (ℓ/min)	Total volume (m^3)	Sampling height (m)
North transect, 0.2 km	A-1	0.2 km north on levee	3317850 m N 763350 m E	23.3	0.64	0.89	1.5
South transect, 1.2 km	A-2	1.2 km south on Texas and Pacific Railroad right-of-way	3315260 m N 762950 m E	24.2	0.69	1.01	1.5
East transect, 0.4 km	A-3	0.4 km east on Moss Lane	3317650 m N 764380 m E	23.0	0.69	0.95	1.5
West transect, 0.2 km	A-4	0.2 km west on Fortier Heights Road	3317220 m N 762870 m E	23.2	0.52	0.72	1.5

Table 32. WATER AND SEDIMENT SAMPLING DATA FOR AMERICAN CYANAMID, NEW ORLEANS, LOUISIANA

Sample No.	Exact location, USGS coordinates		Sample type	Volume collected	Percent moisture by weight
W-1	50 m upstream of Discharge 001	3317850 m N 762940 m E	Grab water	1,000 ml	-
W-2	Midway between Discharge Points 001 and 002	3317910 m N 763330 m E	Grab water	1,000 ml	-
W-3	50 m downstream of Discharge 002	3317970 m N 763560 m E	Grab water	1,000 ml	-
Se-1	50 m upstream of Discharge 001	3317850 m N 762940 m E	Sediment	500 cm^3	47
Se-2	50 m downstream of Discharge 002	3317970 m N 763560 m E	Sediment	500 cm^3	64

Table 33. SOIL SAMPLING DATA FOR AMERICAN CYANAMID NEW ORLEANS, LOUISIANA

Sample No.	Exact location, USGS coordinates		Sample size	Percent moisture
So-1	0.2 km north on levee	3317850 m N 763350 m N	250 cm^3	78
So-2	1.2 km south on Texas and Pacific Railroad right-of-way	3315260 m N 762950 m E	250 cm^3	78
So-3	0.4 km east on Moss Lane	3317650 m N 764380 m E	250 cm^3	70
So-4	0.2 km west on Fortier Heights Road	3317220 m N 762870 m E	250 cm^3	67

Table 34. WEATHER CONDITIONS DURING SAMPLING AT AMERICAN CYANAMID, NEW ORLEANS, LOUISIANA: SOURCE OF DATA, NEW ORLEANS INTERNATIONAL AIRPORT, NEW ORLEANS, LOUISIANA

Time (CDT)	Wind		Precipitation[a/]
	Direction	Speed (knots)	
September 14, 1977			
2000	SW	5	None
2100	0	0	None
2200	0	0	None
2300	S	4	None
2400	0	0	None
September 15, 1977			
0100	0	0	None
0200	0	0	None
0300	0	0	None
0400	0	0	None
0500	0	0	None
0600	0	0	None
0700	0	0	Rain
0800	NE	3	Rain
0900	0	0	Rain
1000	NNW	3	Rain
1100	W	13	Rain
1200	0	0	Rain
1300	WNW	6	Rain
1400	W	5	Rain
1500	W	3	Rain
1600	ENE	4	Rain
1700	E	6	Rain
1800	N	2	None
1900	ENE	6	None
2000	E	6	None
2100	E	9	None
2200	ESE	3	None
2300	0	0	None
2400	E	2	None
September 16, 1977			
0100	0	0	None
0200	ESE	3	None
0300	E	2	None
0400	E	4	None
0500	0	0	None
0600	ESE	2	None
0700	ESE	2	None

a/ Precipitation data from sampling site.

Table 35. ACRYLAMIDE CONCENTRATIONS IN AIR SAMPLES FROM AMERICAN CYANAMID, FORTIER, LOUISIANA

Sample No.	Sampling period (hr)	Volume (m^3)	Type of sample	µg found	µg/m^3
A-1	9/14/77 2025-	0.89	Air[a]	< 0.1	< 0.12[c]
	9/16/77 0538		Particulate[b]	< 0.4	< 0.45[c]
A-2	9/14/77 2145-	1.01	Air[a]	< 0.1	< 0.10
	9/16/77 0658		Particulate[b]	< 0.8	< 0.80
A-3	9/14/77 2100-	0.95	Air[a]	< 0.1	< 0.11
	9/16/77 0625		Particulate[b]	< 0.8	< 0.85
A-4	9/14/77 2045-	0.72	Air[a]	< 0.1	< 0.14
	9/16/77 0609		Particulate[b]	0.8	< 1.12

a/ Chromosorb 101 extract.
b/ Millipore filter extract.
c/ Based on instrumental detection limit and volume of air sampled.

Table 36. ACRYLAMIDE CONCENTRATIONS IN WATER SAMPLES FROM AMERICAN CYANAMID, FORTIER, LOUISIANA

Sample No.	Aliquot volume (ml)	Final volume (ml)	µg found	µg/ℓ
W-1	250	1.0	< 0.2	< 0.8[a/]
W-2	250	1.0	< 0.2	< 0.8
W-3	250	1.0	< 0.2	< 0.8

a/ Based upon instrument detection limit and sample size.

Table 37. ACRYLAMIDE CONCENTRATIONS IN SOIL AND SEDIMENT SAMPLES FROM AMERICAN CYANAMID, FORTIER, LOUISIANA

Sample No.	Sample size wet (g)	Final volume (ml)	µg found	µg/g
So-1	50	1	< 1	< 0.02[a/]
So-2	50	1	< 1	< 0.02
So-3	50	1	< 1	< 0.02
So-4	50	1	< 1	< 0.02
Se-1	50	1	< 1	< 0.02
Se-2	50	1	< 1	< 0.02

a/ Based upon instrumental detection limit and wet sample size.

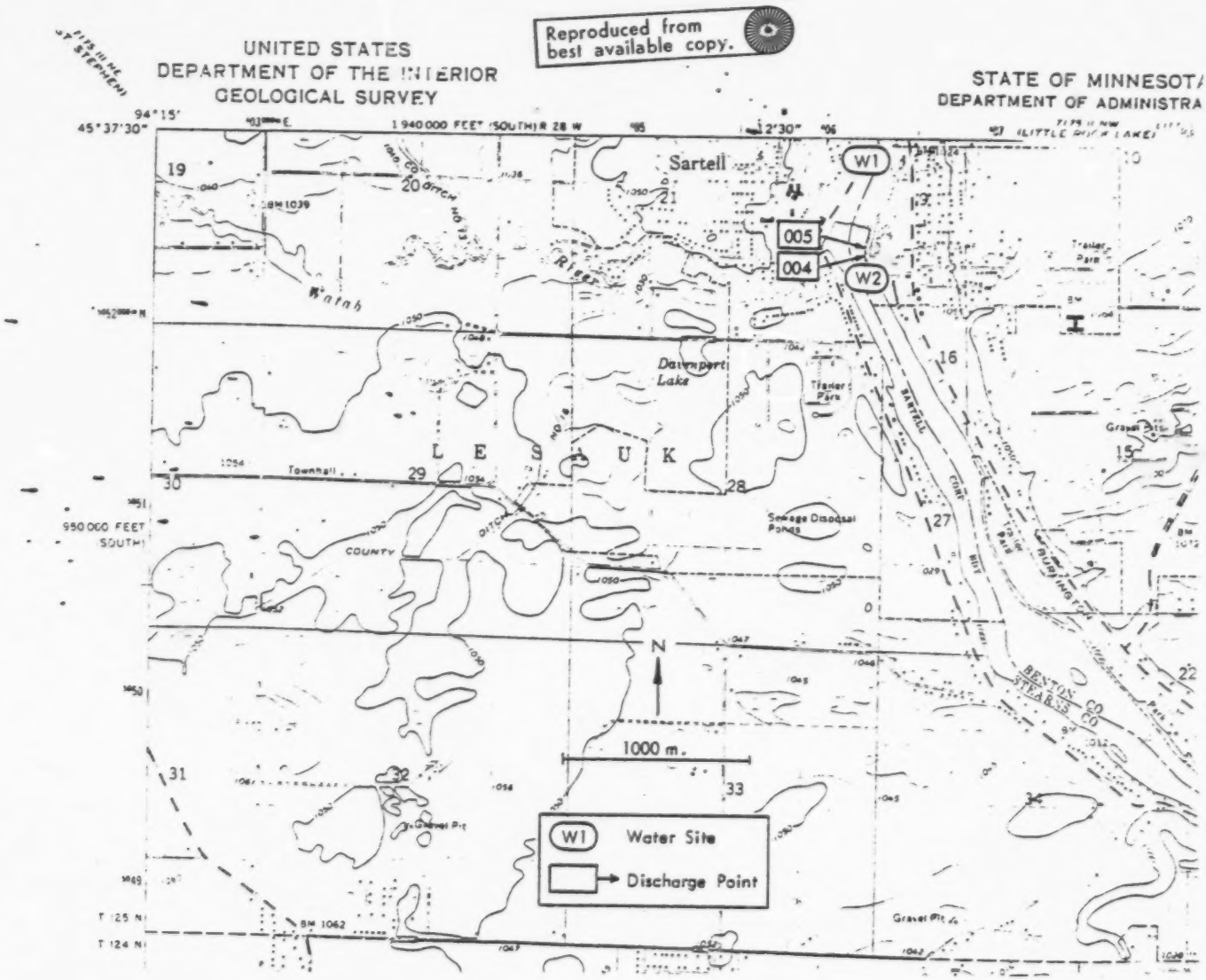

Figure 10. Sampling locations at St. Regis Paper Company, Sartell, Minnesota

Due to the rocky nature of the river bottom, sediment samples were not obtained. Grab water samples were taken on October 6, 1977, from two locations, upstream and downstream from the discharge points, as shown in Figure 10. Water sampling data are given in Table 38.

Sample Analysis

The results of the analysis of the two water samples are given in Table 39. No AA was found above the detection limit of 0.8 μg/liter established using spiked samples.

DOW CHEMICAL, MIDLAND, MICHIGAN

The environmental samples from the Dow plant were collected and analyzed by another contractor,[1/] using in some instances, different methodologies. Some of the results are included in this report so that all of the environmental measurements will be available in a single document.

Grab 1,000 ml water samples were collected from the points indicated in Figure 11. The results of the analyses are given in Table 40. The reported detection limit was 25 ppb.

Air samples were collected at the six stations shown in Figure 11. The results of the analysis for both vapor and particulate AA are given in Table 41. No AA was reported above the 1 $\mu g/m^3$ detection limit.

Soil samples, collected at the six air sampling stations, were analyzed with the results given in Table 42. No AA was reported above 20 ppb.

Table 38. WATER SAMPLING DATA FOR ST. REGIS PAPER COMPANY, SARTELL, MINNESOTA

Sample No.	Exact location, USGS coordinates		Sample type	Volume collected
W-1	0.6 km north on 152 from bridge	5052750 m N 406230 m E	Grab water	1,000 ml
W-2	State Highway 152 bridge over Mississippi River	5052150 m N 406270 m E	Grab water	1,000 ml

Table 39. ACRYLAMIDE CONCENTRATIONS IN WATER SAMPLES FROM ST. REGIS PAPER COMPANY, SARTELL, MINNESOTA

Sampling station	Aliquot volume (ml)	Final volume (ml)	µg found	µg/ℓ
W-1	250	1	< 0.2	< 0.8[a/]
W-2	250	1	< 0.2	< 0.8

a/ Based upon instrumental detection limit and sample size.

Table 40. ANALYTICAL DATA FOR WATER SAMPLES FROM MIDLAND, MICHIGAN[1/]

Sampling station letter	Exact location, USGS coordinates		Acrylamide analysis (ppb)[a/]
A	Tittabawassee River, upstream of chemical plant	4832000 m N 722442 m E	< 25
B	Tittabawassee River, downstream of chemical plant	4827500 m N 726231 m E	< 25
C	Effluent from outfall at Lingle Drain near Dow Corning	4829692 m N 725308 m E	25-125
D	Tap water samples from Holiday Inn		< 25
E	Snow sample taken at air sampling site No. 3	4832910 m N 723591 m E	< 25

a/ Detection limit 25 ppb, quantitation limit 125 ppb.

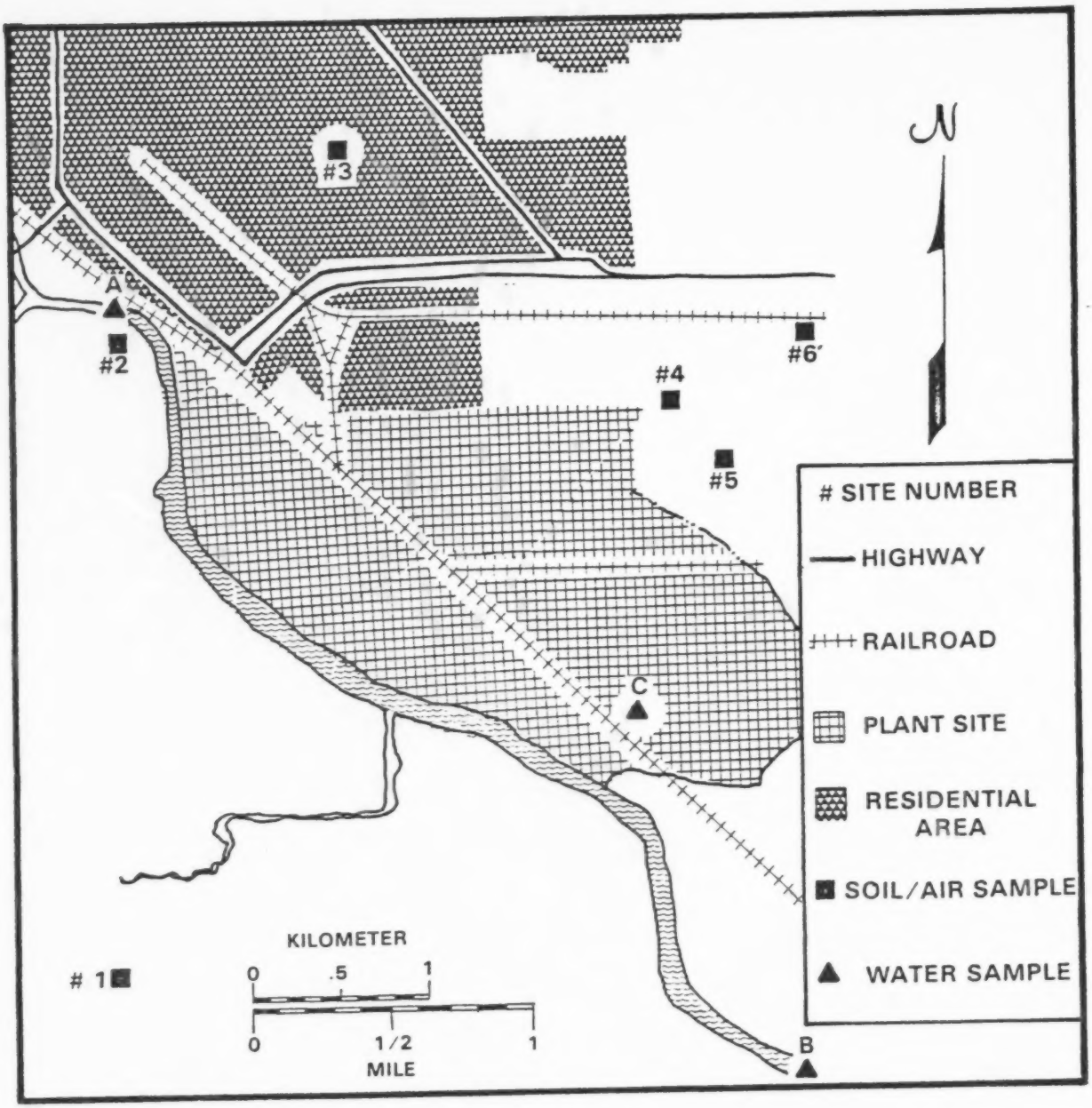

Figure 11. Map of sampling sites for acrylamide at Midland, Michigan

Table 41. ANALYTICAL DATA FOR AIR SAMPLES FROM MIDLAND, MICHIGAN[1/]

Sampling Station	U.S. Geological Survey coordinates	Weather conditions: Wind (av) Direction (degree)	Wind (av) Velocity (m/sec)	Temp. (°F)	General	Sampling period (total 24 hr)		Acrylamide analysis: Particulate[a/] (μg/m³)	Vapor[b/] ppb	Vapor[b/] μg/m³
1	722448 m E	360 (7 hr)	4.80	30-35-25	Partly cloudy	4/6/77	1308-2025	< 1.0	< 1.0	< 1.0
	482819 m N	245 (13 hr)	1.80	25-20-30	Clear		2025-900			
		300 (4 hr)	3.00	30-45	Partly cloudy	4/7/77	900-1323			
2	722417 m E	360 (6 hr)	4.80	31-35-25	Partly cloudy	4/6/77	1408-2025	< 1.0		
	4831905 m N	245 (13 hr)	1.80	25-20-30	Clear		2025-900		< 1.0	< 1.0
		300 (4 hr)	3.00	30-47	Partly cloudy	4/7/77	900-1417			
3	723591 m E	360 (5 hr)	4.40	33-35-25	Partly cloudy	4/6/77	1533-2025			
	4832910 m N	245 (13 hr)	1.80	25-20-30	Clear		2025-900	< 1.0	< 1.0	< 1.0
		300 (7 hr)	3.40	30-49	Partly cloudy	4/7/77	900-1583			
4	725810 m E	360 (3 hr)	3.00	35-25	Partly cloudy	4/6/77	1667-2025		< 1.0	< 1.0
	4831445 m N	245 (13 hr)	1.80	25-20-30	Clear		2025-900	< 1.0		
		315 (8 hr)	3.30	30-46	Partly cloudy	4/7/77	900-1683			
5	725793 m E	360 (2 hr)	1.20	32-25	Partly cloudy	4/6/77	1792-2025		< 1.0	< 1.0
	4831088 m N	245 (13 hr)	1.80	25-20-30	Clear		2025-900	< 1.0		
		330 (9 hr)	3.80	30-38	Partly cloudy	4/7/77	900-1820			
6	726286 m E	360 (8 hr)	5.00	28-35-25	Partly cloudy	4/6/77	1100-2025		< 1.0	< 1.0
	4831833 m N	245 (13 hr)	1.80	25-20-30	Clear		2025-900			
		285 (2 hr)	3.00	30-33	Partly cloudy	4/7/77	900-1122	< 1.0		

a/ Detection limit = 1.0 $\mu g/m^3$.
b/ Detecionn limit = 1.0 ppb.

Table 42. ANALYTICAL DATA FOR SOIL SAMPLES FOR MIDLAND, MICHIGAN

Sampling Station	U.S. Geological Survey coordinates	Acrylamide analysis (ppb)[a]
1	722448 m E 4828198 m N	< 20
2	722417 m E 4831905 m N	
3	723591 m E 4832910 m N	< 20
4	725810 m E 4831445 m N	< 20
5	725793 m E 4831088 m N	< 20
6	726286 m E 4831833 m N	< 20

a/ Detection limit 20 ppb, quantitation limit 100 ppb.

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